

10/634, 516

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NEWS 3 FEB 25 CA/CAPLUS - Russian Agency for Patents and Trademarks
(ROSPATENT) added to list of core patent offices covered
NEWS 4 FEB 28 PATDPAFULL - New display fields provide for legal status
data from INPADO
NEWS 5 FEB 28 BABS - Current-awareness alerts (SDIs) available
NEWS 6 FEB 28 MEDLINE/LMEDLINE reloaded
NEWS 7 MAR 02 GBFULL: New full-text patent database on STN
NEWS 8 MAR 03 REGISTRY/ZREGISTRY - Sequence annotations enhanced
NEWS 9 MAR 03 MEDLINE file segment of TOXCENTER reloaded
NEWS 10 MAR 22 KOREAPAT now updated monthly; patent information enhanced
NEWS 11 MAR 22 Original IDE display format returns to REGISTRY/ZREGISTRY
NEWS 12 MAR 22 PATDPASPC - New patent database available
NEWS 13 MAR 22 REGISTRY/ZREGISTRY enhanced with experimental property tags
NEWS 14 APR 04 EPFULL enhanced with additional patent information and new
fields
NEWS 15 APR 04 EMBASE - Database reloaded and enhanced

NEWS EXPRESS JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT
MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005

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FILE 'HOME' ENTERED AT 11:51:31 ON 14 APR 2005

=> fil reg

COST IN U.S. DOLLARS

SINCE FILE
ENTRY

TOTAL
SESSION

FULL ESTIMATED COST

0.21

0.21

FILE 'REGISTRY' ENTERED AT 11:51:38 ON 14 APR 2005
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Property values tagged with IC are from the ZIC/VINITI data file
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STRUCTURE FILE UPDATES: 13 APR 2005 HIGHEST RN 848462-79-3
DICTIONARY FILE UPDATES: 13 APR 2005 HIGHEST RN 848462-79-3

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 18, 2005

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

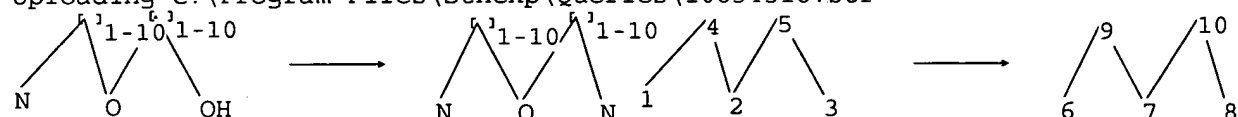
*
* The CA roles and document type information have been removed from *
* the IDE default display format and the ED field has been added, *
* effective March 20, 2005. A new display format, IDERL, is now *
* available and contains the CA role and document type information. *
*

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more
information enter HELP PROP at an arrow prompt in the file or refer
to the file summary sheet on the web at:
<http://www.cas.org/ONLINE/DBSS/registryss.html>

=>

Uploading C:\Program Files\Stnexp\Queries\10634516.str



chain nodes :

1 2 3 4 5 6 7 8 9 10

chain bonds :

1-4 2-4 2-5 3-5 6-9 7-9 7-10 8-10

exact/norm bonds :

1-4 2-4 2-5 3-5 6-9 7-9 7-10 8-10

Match level :

1:CLASS 2:CLASS 3:CLASS 4:CLASS 5:CLASS 6:CLASS 7:CLASS 8:CLASS 9:CLASS
10:CLASS

fragments assigned product role:

containing 6

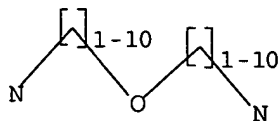
fragments assigned reactant/reagent role:

containing 1

L1 STRUCTURE UPLOADED

=> d query

L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1

SAMPLE SEARCH INITIATED 11:51:52 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 355459 TO ITERATE

0.3% PROCESSED 1000 ITERATIONS 5 ANSWERS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
 BATCH **INCOMPLETE**
PROJECTED ITERATIONS: EXCEEDS 1000000
PROJECTED ANSWERS: EXCEEDS 33017

L2 5 SEA SSS SAM L1

=> logoff y

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	3.01	3.22

STN INTERNATIONAL LOGOFF AT 11:55:36 ON 14 APR 2005

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LOGINID:SSSPTA1204BXD

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TERMINAL (ENTER 1, 2, 3, OR ?):2

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NEWS	3 FEB 25	CA/CAPLUS - Russian Agency for Patents and Trademarks (ROSPATENT) added to list of core patent offices covered
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NEWS EXPRESS JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT
 MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
 AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005

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* * * * * STN Columbus * * * * *

FILE 'HOME' ENTERED AT 11:58:01 ON 14 APR 2005

=> fil casreact		
COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.21	0.21

FILE 'CASREACT' ENTERED AT 11:58:12 ON 14 APR 2005
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FILE CONTENT:1840 - 10 Apr 2005 VOL 142 ISS 15

 *
 * CASREACT now has more than 8 million reactions *
 *

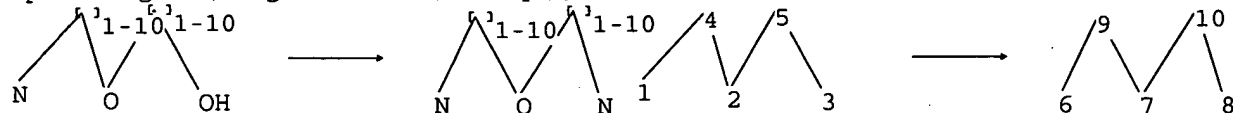
Some CASREACT records are derived from the ZIC/VINITI database (1974-1991) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance

identification.

=>

Uploading C:\Program Files\Stnexp\Queries\10634516.str



chain nodes :

1 2 3 4 5 6 7 8 9 10

chain bonds :

1-4 2-4 2-5 3-5 6-9 7-9 7-10 8-10

exact/norm bonds :

1-4 2-4 2-5 3-5 6-9 7-9 7-10 8-10

Match level :

1:CLASS 2:CLASS 3:CLASS 4:CLASS 5:CLASS 6:CLASS 7:CLASS 8:CLASS 9:CLASS

10:CLASS

fragments assigned product role:

containing 6

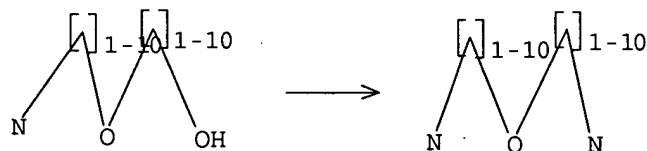
fragments assigned reactant/reagent role:

containing 1

L1 STRUCTURE UPLOADED

=> d query

L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1

SAMPLE SEARCH INITIATED 11:58:32 FILE 'CASREACT'

SCREENING COMPLETE - 42114 REACTIONS TO VERIFY FROM 2677 DOCUMENTS

11.9% DONE 5000 VERIFIED 11 HIT RXNS 2 DOCS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
BATCH **COMPLETE**

PROJECTED VERIFICATIONS: 830550 TO 854010

PROJECTED ANSWERS: 225 TO 845

L2 2 SEA SSS SAM L1 (11 REACTIONS)

=> s l1 full

FULL SEARCH INITIATED 11:58:44 FILE 'CASREACT'

SCREENING COMPLETE - 884836 REACTIONS TO VERIFY FROM 53192 DOCUMENTS

60.0% DONE 530577 VERIFIED 2086 HIT RXNS (4 INCOMP) 126 DOCS

67.8% DONE 600000 VERIFIED 2380 HIT RXNS (4 INCOMP) 147 DOCS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.28

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
BATCH **COMPLETE**

PROJECTED VERIFICATIONS: 884836 TO 884836
PROJECTED ANSWERS: 198 TO 290

L3 147 SEA SSS FUL L1 (2380 REACTIONS)

=> fil caplus

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	106.96	107.17

FILE 'CAPLUS' ENTERED AT 11:59:25 ON 14 APR 2005
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FILE COVERS 1907 - 14 Apr 2005 VOL 142 ISS 16
FILE LAST UPDATED: 13 Apr 2005 (20050413/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l3

L4 147 L3

=> s catalyst

685195 CATALYST
688968 CATALYSTS

L5 878410 CATALYST
(CATALYST OR CATALYSTS)

=> s cu or copper

776648 CU
4633 CUS
778651 CU
(CU OR CUS)

839128 COPPER
422 COPPERS
839193 COPPER
(COPPER OR COPPERS)

```

L6      1116901 CU OR COPPER

=> s zn or zinc
      450419 ZN
      26099 ZNS
      470391 ZN
          (ZN OR ZNS)
      547748 ZINC
      97 ZINCS
      547768 ZINC
          (ZINC OR ZINCS)
L7      730932 ZN OR ZINC

=> s 15 and 16 and 17
L8      14226 L5 AND L6 AND L7

=> s 14 and 18
L9      0 L4 AND L8

=> s "ether alcohol"
      459521 "ETHER"
      141301 "ETHERS"
      517692 "ETHER"
          ("ETHER" OR "ETHERS")
      222713 "ALCOHOL"
      153483 "ALCOHOLS"
      348022 "ALCOHOL"
          ("ALCOHOL" OR "ALCOHOLS")
      551091 "ALC"
      183287 "ALCS"
      644698 "ALC"
          ("ALC" OR "ALCS")
      769293 "ALCOHOL"
          ("ALCOHOL" OR "ALC")
L10     2211 "ETHER ALCOHOL"
          ("ETHER" (W) "ALCOHOL")

=> s 18 and 110
L11     8 L8 AND L10

=> d 111 1-8 abs ibib

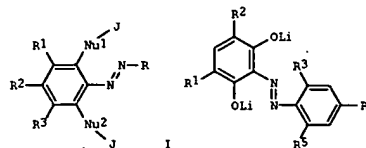
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L11 ANSWER 1 OF 8 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB A method for converting amino ether alcs. (e.g., Me₂NCH₂CH₂OCH₂CH₂OH) to amino ether amines (e.g., Me₂NCH₂CH₂OCH₂CH₂NHMe₂) by amination with an alkylamine (e.g., dimethylamine) in the vapor phase with a catalyst comprising copper and zinc.
 The catalyst may also comprise an alkali metal, an alkaline earth metal, or a lanthanide element as a promoter.

ACCESSION NUMBER: 2005:116082 CAPLUS
 DOCUMENT NUMBER: 142:176435
 TITLE: Vapor-phase process for the catalytic amination of amino ether alcohols into amino ether amines
 INVENTOR(S): Mitchell, John William; Underwood, Richard Paul; Ford, Michael Edward; Vedage, Gamin Ananda; Li, Hong-Xin
 PATENT ASSIGNEE(S): Air Products and Chemicals, Inc., USA
 SOURCE: Eur. Pat. Appl., 12 pp.
 CODEN: EPXKXW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1505059	A1	20050209	EP 2004-18378	20040803
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR				
US 2005033091	A1	20050210	US 2003-634516	20030804
JP 2005033913	A2	20050303	JP 2004-228411	20040804
PRIORITY APPLN. INFO.: 4			US 2003-634516	A 20030804
REFERENCE COUNT:			THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT	

L11 ANSWER 2 OF 8 CAPLUS COPYRIGHT 2005 ACS ON STN
 G1



AB Azo complexes of 4-12 B group metals used as catalysts for (co)polymerization of olefins are prepared by mixing solns. of azo compds.

I (Nu1 and Nu2 = O, S, Se, PRA, NRA or COO, Ra = H or optionally substituted alkyl or aryl group, J = A group 1-2 element, R, R1, R2 and R3 = H, halogen, optionally substituted C1-8 alkyl, C2-8 alkenyl, C3-12 cycloalkyl, C7-13 arylalkyl or C6-14 aryl, R1R2 and R2R3 can form rings) and M(L1)(L2)(L3)z (M = a 4-12 side group metal, L1 = neutral ligand, such as phosphines, amines, tetraaryllalkyl ethylenediamine, ethers, alcs., derivs. of pyridine, CO, C1-12 alkyl nitrile, C6-14 aryl nitrile, L2 and L3 = anionic ligands, such as halide-, amide-, C1-6 alkyl-, allyl-, methallyl-, benzyl- or aryl-anions, w, y and z = 0-3) in polar solvents. Thus, mixing a solution of II (R1 = R2 = tert-Bu, R3 = R5 = iso-Pr, R4 = H) in a mixture of toluene/chlorobenzene with a solution of bis(1,5-cyclooctadiene)nickel in toluene gave a catalyst used in polymerization of ethylene in autoclave at 8 bar and 30-60° in the presence of a mixture of BF₃/diethyl ether as activator..

ACCESSION NUMBER: 2004:753180 CAPLUS
 DOCUMENT NUMBER: 141:261201
 TITLE: Method of preparing of olefin polymerization catalyst containing azo group
 INVENTOR(S): Weiss, Thomas
 PATENT ASSIGNEE(S): Bayer A.-G., Germany
 SOURCE: Eur. Pat. Appl., 25 pp.
 CODEN: EPXKXW
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1457502	A1	20040915	EP 2004-4501	20040227
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
DE 10310289	A1	20040923	DE 2003-10310289	20030310
US 2004181018	A1	20040916	US 2004-793512	20040304
CA 2459658	AA	20040910	CA 2004-2459658	20040305
JP 2004269889	A2	20040930	JP 2004-63842	20040308
PRIORITY APPLN. INFO.: 5			DE 2003-10310289	A 20030310
OTHER SOURCE(S): MARPAT 141:261201				
REFERENCE COUNT:			THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT	

L11 ANSWER 2 OF 8 CAPLUS COPYRIGHT 2005 ACS ON STN (Continued)

L11 ANSWER 3 OF 8 CAPLUS COPYRIGHT 2005 ACS ON STN

AB A process of converting CO and CO₂ present in feed gas of ammonia synthesis comprising H and N as major components into useful coproducts is disclosed. Firstly, the alc.-ether formed reaction is carried out by using copper group catalyst, and the alc.-hydrocarbon formed reaction is sep. preformed by using iron group catalyst. The alc.-ether and alc.-hydrocarbon products are separated by water cooling and condensing, and discharged into corresponding storage tanks, and the remaining gas, in which the amount of CO and CO₂ is ≤10 ppm, is fed to ammonia synthesis system. Since the alc.-hydrocarbon can be used as liquid fuel, the product composition of ammonia plant is improved, and the amount of CH₄ fed into ammonia synthesis system is decreased, and the amount of venting is thus reduced.

ACCESSION NUMBER: 2004:718496 CAPLUS
 DOCUMENT NUMBER: 141:227255
 TITLE: A joint process for preparing alcohol-ether, alcohol-hydrocarbon and synthesis ammonia
 INVENTOR(S): Xie, Din Zhong; Dai, Feng Yu; Feng, Yong; Li, Chun Yang
 PATENT ASSIGNEE(S): Peop. Rep. China
 SOURCE: PCT Int. Appl., 21 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: Chinese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004074226	A1	20040902	WO 2003-CN136	20030219
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MN, MW, MX, MY, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
PRIORITY APPLN. INFO.: 3			WO 2003-CN136	20030219
REFERENCE COUNT:			THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT	

L11 ANSWER 4 OF 8 CAPLUS COPYRIGHT 2005 ACS on STN
AB The carbonyl compds. are prepared by treatment of unsaturated ethers with alcs. in the presence of Group 4-12 metals and H₂O. Thus, 55:45 mixture of 3-methoxy-1-butene and 1-methoxy-2-butene 1, MeOH 2, and H₂O 1.30 mol were passed through a reactor containing Pd/C at 150° over 3 h to give 46.9% MEK and 16.1% butanal.

ACCESSION NUMBER: 2004:134046 CAPLUS
DOCUMENT NUMBER: 140:163467
TITLE: Preparation of ketones and/or aldehydes from unsaturated ethers and alcohols
INVENTOR(S): Kobayashi, Kenji; Asano, Satoshi; Taki, Takayuki; Suzuki, Takahiro; Saito, Toshiya; Takatsuna, Kazutoshi
PATENT ASSIGNER(S): Tonen Chemical Corp., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 11 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2004051518	A2	20040219	JP 2002-209593	20020718
PRIORITY APPLN. INFO.:			JP 2002-209593	20020718

L11 ANSWER 5 OF 8 CAPLUS COPYRIGHT 2005 ACS on STN
AB This invention includes catalysts comprising rhenium (atomic number 75), nickel, cobalt, boron, and copper and/or ruthenium impregnated on a support material and a process for preparing said catalyst, said process comprising (i) impregnating a mixture of metals comprising rhenium, cobalt, copper and/or ruthenium, boron and nickel on a support material selected from the group consisting of alpha-alumina, silica, silica-alumina, kieselguhrs or diatomaceous earths, and silica-titanias; and (ii) activating said catalyst by heating the catalyst in the presence of hydrogen at an effective temperature preferably in the range of 150°C to 500°C for a sufficient period preferably of from 30 min to 6 h. A further feature of the present invention was a method for producing amine products by the catalytic amination of alkane or arylalkane derivs. including epoxides, alcs., diols, polyethers, polyols, alc. amines, ketones, imino compds., iminoalcs., ether alcs., and mixts. thereof, said process comprising contacting said lower alkane or arylalkane derivs. with ammonia and/or reactant amine at an effective temperature preferably

from 150°C to 500°C and in the presence of hydrogen and the catalyst as described hereinabove. These catalysts exhibit high conversion and selectivity in the amination.
ACCESSION NUMBER: 1997:97181 CAPLUS
DOCUMENT NUMBER: 126:105750
TITLE: Supported metal catalyst for amination
INVENTOR(S): Chang, Dane; Sherrod, Fred A.
PATENT ASSIGNER(S): Dow Chemical Company, USA; Chang, Dane; Sherrod, Fred A.
SOURCE: PCT Int. Appl., 44 pp.
CODEN: PIXKX2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9638226	A1	19961205	WO 1996-US7876	19960529
W: BR, CA, CN, JP, KR, US				
RS: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
US 5817593	A	19981006	US 1995-45982	19950602
CA 2223036	AA	19961205	CA 1996-2223036	19960529
EP 828558	A1	19980318	EP 1996-920544	19960529
EP 828558	B1	20011205		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
CN 1186453	A	19980701	CN 1996-194385	19960529
CN 1086314	B	20020619		
BR 9608921	A	19990302	BR 1996-8921	19960529
JP 2001501524	T2	20010206	JP 1996-536609	19960529
AT 209962	E	20011215	AT 1996-920544	19960529
US 5952529	A	19990914	US 1998-105568	19980622
PRIORITY APPLN. INFO.:			US 1995-45982	A 19950602
			WO 1996-US7876	W 19960529

L11 ANSWER 6 OF 8 CAPLUS COPYRIGHT 2005 ACS on STN
AB A process for the preparation of improved active Cu catalysts, useful for nitrile hydration to amides, by the reduction of high valence anhydrous Cu salts comprises reducing 21 high valence anhydrous Cu salts having the formula Cu_aX_b, where X is an anion, such as a halide, sulfate or nitrate, a is 1 or 2, and b is 1 or 2, with an activated metal having low oxidation potential and selected from Groups IA, IIA or IIB in a solvent selected from ethers, alcs., or mixts. thereof at a temperature of 30-80° and under an inert atmospheric

ACCESSION NUMBER: 1990:521488 CAPLUS
DOCUMENT NUMBER: 113:121488
TITLE: Process for the preparation of copper catalysts
INVENTOR(S): Ravindranathan, Marayil; Sivaram, Swaminathan
PATENT ASSIGNER(S): Indian Petrochemicals Corp. Ltd., India
SOURCE: Indian, 25 pp.
CODEN: INOXAP
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
IN 164875	A	19890624	IN 1986-BO154	19860526
PRIORITY APPLN. INFO.:			IN 1986-BO154	19860526

L11 ANSWER 7 OF 8 CAPLUS COPYRIGHT 2005 ACS on STN
G1 For diagram(s), see printed CA issue.
AB 1,4-Endomethylenetetrahydrofluorene, (I) (prepared from cyclopentadiene and indene, cf. C.A. 32, 3368.9) is treated in the presence of an acidic condensing agent with an organic compound having at least one OH group. A simultaneous rearrangement accompanies the reaction and the product is an ether having an endoethylenecyclopentanoidanyl (R') group, C14H15. Thus of the two isomers A is regarded as more probable. With polyhydric compds., I can react with each OH group. To a stirred solution of 60 g. ethylene chlorohydrin and 5 g. BF₃·OEt₂ at 70° there was added gradually 91 g. of I. The mixture was heated at 95° for 5 h. The dark oil was washed with dilute NaOH, water, dried, and distilled in vacuo to give 102 g. ClCH₂CH₂OR'', colorless oil, b₁ 157°. The Cl atom is readily replaceable and the compound is thus valuable as an intermediate for the preparation of insecticides. Trimethylene chlorohydrin and I gave Cl(CH₂)₃OR''. The following compds. were prepared from the appropriate alc. and I by using the BF₃·OEt₂ complex or the corresponding Et₂O complex as a catalyst: Br(CH₂)₃OR'', b₀ 5 168-73°; NCH₂CH₂OR'', colorless oil, b₂ 174-6°; HOCH₂CH₂OR'', colorless oil, b₁ 157-9°; R''OCH₂CH₂OR'', b₁ 275°; HOCH₂CH₂OC(CH₂)₂OR'', colorless oil, b₀ 5 175-7°; HOCH₂CH₂(OH)CH₂OR'', viscous oil, b₀ 5 200°; EtOOCCH(CH₃)OR'', pale yellow oil, b₀ 5 165-70° which on hydrolysis gave the corresponding acid (II) whose Cu, Hg, and Bi salts are soluble in oil and possess fungicidal activity. The Pb, Co, Zn, and Mn salts of II are of value as siccatives for paints and varnishes. O₂NCH₂CH₂OR'', pale yellow oil, b₁ 183-7°; C₆H₁₁OR'', colorless oil, b₀ 5 164-6°; PhCH₂OR'', colorless crystalline compound, b₁ 195°; ClCH₂CH₂OC(CH₂)₂OR'', nearly colorless oil, b₀ 5 170-3°; BuEt-CHCH₂OR'', colorless oil, b₁ 173-6°; CH₃(CH₂)₁₀CH₂OR'', yellow oil, b₁ 219-24°; CH₂CHCH₂OR'', colorless oil, b₇ 167-70°; CH₂CH₂CH₂CHOR'', colorless oil, b₀ 5 169-73°; EtOCH₂CH₂OR'', colorless oil, b₁ 155-8°; PhOCH₂CH₂OR'', colorless oil, b₁ 213-16°; EtOOCCH₂OR'', colorless oil, b₁ 165-70°; NCSCH₂CH₂OC(CH₂)₂OR'', pale yellow oil, b₁ 215-220°; ClCH₂C(CH₃)₂OR'', colorless oil, b₁ 2 152-8°; BuOCH₂CH₂OC(CH₂)₂OR'', colorless oil, b₁ 192-6°; ClCH₂CH(OH)CH₂OR'', colorless oil, b₁ 190-5°. With a catalyst prepared from cold MeOH and H₂SO₄, MeOR'' was prepared by refluxing the alc. with I. It was a colorless oil, b₆ 147-9°. Ethanol and I in the presence of ethylsulfuric acid gave EtOR'', b₆ 152-5°.

ACCESSION NUMBER: 1945:20765 CAPLUS
DOCUMENT NUMBER: 39:20765
ORIGINAL REFERENCE NO.: 39:3300a-i
TITLE: Ethers of endoethylenecyclopentanoidan
INVENTOR(S): Bruson, Herman A.
PATENT ASSIGNER(S): The Resinous Products & Chemical Co., Inc.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2376865		19450529	US	

L11 ANSWER 8 OF 8 CAPLUS COPYRIGHT 2005 ACS on STN
 AB These are produced by hydrogenating the corresponding dinitrile containing
 6-10 C atoms in which the nitrile groups are separated by at least 4 C
 atoms,
 the reduction being effected in the liquid phase at 25-200° under
 pressure in the presence of NH3 and a hydrogenating catalyst. A
 solvent may be present, e. g., an ether, alc. or
 hydrocarbon. Pyrophoric or stabilized Ni catalysts in the form
 of powder or supported on powdered materials such as kieselguhr or pumice,
 or
 other metals or Cu or Zn compds. may be used as
 catalysts. Among examples, hexamethylenediamine is prepared in the
 presence of NH3 from adiponitrile with use of a Ni catalyst both
 with and without the presence of MeOH.
 ACCESSION NUMBER: 1939:6520 CAPLUS
 DOCUMENT NUMBER: 33:6520
 ORIGINAL REFERENCE NO.: 33:993b-d
 TITLE: Aliphatic diamines
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 490922		19380816	GB	

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=> s "ether amine"
      459521 "ETHER"
      141301 "ETHERS"
      517692 "ETHER"
          ("ETHER" OR "ETHERS")
      254291 "AMINE"
      240456 "AMINES"
      389613 "AMINE"
          ("AMINE" OR "AMINES")
L12      871 "ETHER AMINE"
          ("ETHER" (W) "AMINE")

=> d his

      (FILE 'HOME' ENTERED AT 11:58:01 ON 14 APR 2005)

      FILE 'CASREACT' ENTERED AT 11:58:12 ON 14 APR 2005
L1          STRUCTURE UPLOADED
L2          2 S L1
L3          147 S L1 FULL

      FILE 'CAPLUS' ENTERED AT 11:59:25 ON 14 APR 2005
L4          147 S L3
L5          878410 S CATALYST
L6          1116901 S CU OR COPPER
L7          730932 S ZN OR ZINC
L8          14226 S L5 AND L6 AND L7
L9          0 S L4 AND L8
L10         2211 S "ETHER ALCOHOL"
L11         8 S L8 AND L10
L12         871 S "ETHER AMINE"

=> s l12 and l8
L13         2 L12 AND L8

=> s l13 not l11
L14         1 L13 NOT L11

=> d l14 abs ibib

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L14 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Perylenetetracarboxylic diimides with a crown ether group connected to
 ≥1 N atom are obtained from crown ether amine
 derivs. and the appropriate perylenetetracarboxylic derivative The dyes
 have
 the ability to complex with metals, forming strongly fluorescing complexes
 and thus may be used for fluorimetric determination of metal ions. Thus,
 2-(aminomethyl)-15-crown-5 was condensed with N-(1-hexylheptyl)perylene-
 3,4,9,10-tetracarboxylic acid-3,4-dianhydride-9,10-imide to give a
 fluorescent dye with a 1-hexylheptyl group and a 2-methylene-15-crown-5
 group. This dye formed fluorescent complexes with Fe and other metals.
 ACCESSION NUMBER: 1998:603279 CAPLUS
 DOCUMENT NUMBER: 129:246520
 TITLE: Perylene crown ether fluorescent dyes, their
 preparation and their use as fluorescent complex
 formers for metallic materials
 INVENTOR(S): Langhals, Heinz; Jona, Wolfgang
 PATENT ASSIGNEE(S): Germany
 SOURCE: Ger. Offen., 32 pp.
 CODEN: GWCKEX
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 2
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19709008	A1	19980910	DE 1997-19709008	19970305
WO 9839333	A1	19980911	WO 1998-EP1023	19980223
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, AM, AZ, BY, EG, KG, KZ, MD, RU, TJ, TM RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
AU 9867237	A1	19980922	AU 1998-67237	19980223
EP 966468	A1	19991229	EP 1998-912370	19980223
EP 966468	B1	20040922		
R: CH, DE, FR, GB, IT, LI				
JP 2001518078	T2	20011009	JP 1998-538112	19980223
PRIORITY APPLN. INFO.:				
			DE 1997-19709004	A 19970305
			DE 1997-19709008	A 19970305
			WO 1998-EP1023	W 19980223
OTHER SOURCE(S): MARPAT 129:246520				

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=> s ?alcohol
      225428 ?ALCOHOL
      551091 ALC
      183287 ALCS
      644698 ALC
            (ALC OR ALCS)
L15      730729 ?ALCOHOL
            (?ALCOHOL OR ALC)

=> s ?amine
L16      1405122 ?AMINE

=> s l15 and l16
L17      94939 L15 AND L16

=> d his
      (FILE 'HOME' ENTERED AT 11:58:01 ON 14 APR 2005)

      FILE 'CASREACT' ENTERED AT 11:58:12 ON 14 APR 2005
L1      STRUCTURE UPLOADED
L2      2 S L1
L3      147 S L1 FULL

      FILE 'CAPLUS' ENTERED AT 11:59:25 ON 14 APR 2005
L4      147 S L3
L5      878410 S CATALYST
L6      1116901 S CU OR COPPER
L7      730932 S ZN OR ZINC
L8      14226 S L5 AND L6 AND L7
L9      0 S L4 AND L8
L10     2211 S "ETHER ALCOHOL"
L11     8 S L8 AND L10
L12     871 S "ETHER AMINE"
L13     2 S L12 AND L8
L14     1 S L13 NOT L11
L15     730729 S ?ALCOHOL
L16     1405122 S ?AMINE
L17     94939 S L15 AND L16

=> s l17 and l8
L18     257 L17 AND L8

=> s l18 not l11
L19     253 L18 NOT L11

=> s l19 not l14
L20     253 L19 NOT L14

=> d l20 1-253 abs ibib

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L20 ANSWER 1 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB The present invention provides a precious metal-metal oxide composite cluster, wherein said cluster is formed as a single particle by combining a precious metal portion comprising a single atom or an aggregate of a plurality of atoms consisting of one or more precious metals, and a metal oxide portion comprising a single mol. or an aggregate of a plurality of mols. consisting of one or more metal oxides, and wherein said particle has a particle size between 1 and 100 nm.

ACCESSION NUMBER: 2005:259621 CAPLUS
DOCUMENT NUMBER: 142:304316
TITLE: Precious metal-metal oxide composite cluster
INVENTOR(S): Okubo, Kyoko
PATENT ASSIGNEE(S): Tanaka Kikinzoku Kogyo K.K., Japan
SOURCE: U.S. Pat. Appl. Publ., 7 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2005065026	A1	20050324	US 2004-938669	20040910
EP 1522341	A1	20050413	EP 2004-255748	20040922
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, HK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR				
PRIORITY APPLN. INFO.:				JP 2003-329784 A 20030922

L20 ANSWER 2 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB The invention relates to a multistage process for continuous and phosgene-free preparation of cycloaliph. diisocyanates. Thus, perhydrogenated diphenylmethanediamine 263.0, urea 154.5, and n-butanol 555.9 g were fed into a distillation reactor every hour and boiled at 135° with average residence time 8 h while continuously removing ammonia released at atmospheric

pressure, the bisurea butanol solution obtained in the bottom of the reactor was preheated at 190°, conducted to the uppermost tray of the reactor, and converted at from 11 to 14 bar at 220°, 506.8 g/h butanol was fed into the bottom of the reactor while removing alc. and ammonia at the top of the reactor, the reactor effluent was evaporated and the remaining 628.7 g/h bis(4-butoxycarbonylaminoethyl)methane was conducted as a melt into the circulation of the falling-film evaporator of the cleavage and rectification column, and the deblocking reaction was carried out at 237° in the presence of 18 pm steady state concentration tin chloride, the cleavage gases, dicyclohexylmethane diisocyanate, and butanol were condensed and purifying distilled to give pure dicyclohexylmethane diisocyanate with purity >99.5%.

ACCESSION NUMBER: 2005:160891 CAPLUS
DOCUMENT NUMBER: 142:261907
TITLE: Multistage continuous preparation of cycloaliphatic diisocyanates
INVENTOR(S): Kohlstruk, Stephan; Kreczinski, Manfred; Elm, Rainer; Michalczak, Hans-Werner
PATENT ASSIGNEE(S): Degussa A.-G., Germany
SOURCE: U.S. Pat. Appl. Publ., 9 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2005043563	A1	20050224	US 2004-922910	20040823
DE 10338509	A1	20050317	DE 2003-10338509	20030822
EP 1512682	A1	20050309	EP 2004-103133	20040702
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, HK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR				
CA 2478743	AA	20050222	CA 2004-2478743	20040820
JP 2005068148	A2	20050317	JP 2004-240675	20040820
PRIORITY APPLN. INFO.:				DE 2003-10338509 A 20030822

L20 ANSWER 3 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB Title comds. are prepared by reaction of alkyleneopolyamines with C22 alkyl alcs. in the presence of Cu-containing oxide catalysts. Ethylenediamine was alkylated by EtOH in the presence of CuO/Al2O3 catalyst under 1 MPa H at 200° for 8 h to give N-ethylethylenediamine with 59.7% selectivity at 74.4% conversion.

ACCESSION NUMBER: 2005:135634 CAPLUS
DOCUMENT NUMBER: 142:218956
TITLE: Preparation of N-monoalkyl alkyleneopolyamines
INVENTOR(S): Yamakawa, Akira; Ogawa, Tsukasa
PATENT ASSIGNEE(S): Tosoh Corp., Japan; Sagami Chemical Research Center
SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.
CODEN: JXOXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2005041806	A2	20050217	JP 2003-202220	20030728
PRIORITY APPLN. INFO.:				JP 2003-202220 20030728
OTHER SOURCE(S): CASREACT 142:218956				

L20 ANSWER 4 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB Title composition comprises a curable resin having a silicone group SIXIXX3 or

SIXIXX2 and a Lewis acid curing catalyst selected from metal halides and boron halides or a complex of the Lewis acid, wherein X1, X2, X3 = hydrolyzable group and R1 = C1-20 (un)substituted organic group. When the silicone group is SIXIXX2, the curable resin further contains a polar element which is a urethane bond, thiourethane bond, urea bond, thiourea bond, substituted urea bond, substituted thiourea bond, amide bond, sulfide bond, hydroxy, secondary amino, or tertiary amino. The ingredients can be formulated so as to constitute a two-pack type adhesive in which the curable resin and the catalyst for solidification are sep. packed. Thus, 100.0 g SDX 1690 an allyl-terminated polyether and 10.3 g DMDS 1,5-dimercapto-3-thiapentane were heated at 90°, 0.5 g ALEN was and 15.6 g KBM 5103 was added therein and reacted at 85° for 3 h to give a trimethoxysilyl-containing curable resin, 100 parts of which

was mixed with 3 parts boron trifluoride ethylamine complex, showing fast curing time at room temperature

ACCESSION NUMBER: 2005:74157 CAPLUS
DOCUMENT NUMBER: 142:157362
TITLE: Moisture-curable resin composition with fast curing speed for room temperature curable adhesive
INVENTOR(S): Mori, Shigeki; Nomura, Yukihiko; Iyo, Kazuhiro; Sato, Shinichi
PATENT ASSIGNEE(S): Konishi Co., Ltd., Japan
SOURCE: PCT Int. Appl., 225 pp.
CODEN: PIXX2
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005007751	A1	20050127	WO 2004-JP10549	20040716
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, BG, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
JP 2005054174	A2	20050303	JP 2004-208672	20040715
PRIORITY APPLN. INFO.:				JP 2003-277042 A 20030718
REFERENCE COUNT: 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT				

L20 ANSWER 5 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Supported reactive catalysts having a controlled coordination structure and methods for their production are disclosed. The supported catalysts of the present invention are useful for the preparation of hydrogen peroxide with high selectivity in addition to other chemical conversion reactions. The supported catalyst comprises catalyst particles having top or outer layer of atoms in which at least a portion of the atoms exhibit a controlled coordination number of 2. The catalyst and methods may be used for the concurrent in situ and ex situ conversion of organic compounds. In addition, a process is provided for catalytically producing hydrogen peroxide from hydrogen and oxygen feeds by contacting them with the catalysts of the invention and a suitable organic liquid solvent having a Solvent Selection Parameter (SSP) between 0.14+10⁻⁴ and 5.0+10⁻⁴.

ACCESSION NUMBER: 2005:59954 CAPLUS
 DOCUMENT NUMBER: 142:101067
 TITLE: Supported catalysts having a controlled coordination structure and methods for preparing such catalysts
 INVENTOR(S): Zhou, Bing; Rueter, Michael; Parasher, Sukesh
 PATENT ASSIGNEE(S): USA
 SOURCE: U.S. Pat. Appl. Publ., 28 pp.
 CODEN: USXXCO
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 2
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2005014635	A1	20050120	US 2003-618808	20030714
WO 2005009611	A2	20050203	WO 2004-US19439	20040617
WO 2005009611	A3	20050317		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZH, ZW
 RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZH, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.:
 US 2003-618808 A 20030714
 US 2003-618909 A 20030714

L20 ANSWER 6 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Nanostructured carbon materials having excellent crystallinity and large surface area are prepared by formation of nanostructured carbon material-metal-inorg. oxide composite through catalytic graphitization of a polymeric carbon precursor-metal salt-inorg. oxide composite, removal of inorg. oxide using an etchant, and removal of metal through an acid treatment, wherein an inorg. oxide material is added in the reaction mixture to increase the surface area of the nanostructured carbon material, and the metal salt is used as a graphitization catalyst. The polymeric carbon precursors can be resorcinol-formaldehyde-gel (RF-gel), phenol-formaldehyde-gel, phenol resin, melamine-formaldehyde-gel, poly(furfurylalc.), poly(acrylonitrile), sucrose, polypyrrole, polydivinylbenzene, or petroleum pitch. The metal salt can be an acetate, acetyl acetate, fluoride, chloride, bromide, nitrate, sulfate, phosphate, oxalate, perchlorate, or alkoxides of Fe, Co, Ni, Mo, V, Y, Zr, Nb, Li, Mg, Al, Si, K, Ca, Ti, Cr, Mn, Cu, Zn, Ga, Ge, As, In, Sn, Sb, La, Hf, Ta, or W. The inorg. oxide can be silica, alumina, titania, ceria, zirconia, tin oxide, or yttria. The etchant can be HF, NaOH, KOH, Mg(OH)₂, Ca(OH)₂, or LiOH. The resultant nanostructured carbon materials possess the characteristics of excellent crystallinity and large surface area (> 200 m²/g), where such characteristics are well suited for low temperature fuel cell electrode applications.

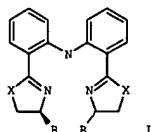
ACCESSION NUMBER: 2005:34425 CAPLUS
 DOCUMENT NUMBER: 142:96854
 TITLE: Manufacture of nanostructured carbon materials having excellent crystallinity and large surface area suitable for fuel cell electrodes
 INVENTOR(S): Hyeon, Taegwhan; Han, Sangjin
 PATENT ASSIGNEE(S): Seoul National University, S. Korea
 SOURCE: U.S. Pat. Appl. Publ., 15 pp.
 CODEN: USXXCO
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2005008562	A1	20050113	US 2003-658586	20030908
WO 2005005471	A1	20050120	WO 2003-KR1377	20030710

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZH, ZW
 RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZH, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.:
 WO 2003-KR1377 A 20030710

L20 ANSWER 7 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 GI



AB A series of novel C2-sym. bis(thiazoline) ligands I (X = S; R = i-Pr, i-PrCH₂, Ph, Et, t-Bu) with a diphenylamine backbone as a linkage between two thiazoline rings were synthesized by the use of the simple reagent phosphorus pentasulfide. Their application in the catalytic asym. Henry reaction of α -keto esters was investigated with comparison to bis(oxazoline) ligands I (X = O). Cu (II)-bis(oxazoline) complexes furnished moderate enantioselectivities, while Cu(II)-bis(thiazoline) complexes gave higher enantioselectivities with neat nitromethane. The enantioselectivity was improved when a halogenated solvent, such as CH₂Cl₂ was used, but the yield obtained lower than that in neat reactions.

ACCESSION NUMBER: 2004:920922 CAPLUS
 DOCUMENT NUMBER: 142:74494
 TITLE: Facile synthesis of C2-symmetric tridentate bis(thiazoline) and bis(oxazoline) ligands and their application in the enantioselective Henry reaction
 AUTHOR(S): Lu, Shao-Feng; Du, Da-Ming; Zhang, Shi-Wei; Xu, Jiaxi
 CORPORATE SOURCE: College of Chemistry and Molecular Engineering, Key Laboratory of Bioorganic Chemistry and Molecular Engineering of Ministry of Education, Peking University, Beijing, 100871, Peop. Rep. China
 SOURCE: Tetrahedron: Asymmetry (2004), 15(21), 3433-3441
 CODEN: TASYE3; ISSN: 0957-4166
 PUBLISHER: Elsevier B.V.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 REFERENCE COUNT: 93
 THERE ARE 93 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 8 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Catalyst is one of key issues during amination of fatty alc. with dimethylamine for preparation of long chain alkyl di-Me tertiary amine. The effects on selectivity, activity, BET surface area, and average pore diameter of catalyst by adding La, Ba, or Zn to the system of Cu-Ni supported on CaCO₃ were studied. The selectivity of Cu-Ni catalyst was improved effectively by addition of La, Ba, and Zn, but the BET surface area and average pore diameter of catalyst were declined.

ACCESSION NUMBER: 2004:861469 CAPLUS
 TITLE: Study of catalysts for amination of fatty alcohol
 AUTHOR(S): Tan, Ping; Li, Qixiao
 CORPORATE SOURCE: China Research Institute of Daily Chemical Industry, Taiyuan, 030001, Peop. Rep. China
 SOURCE: Ruyong Huaxue Gongye (2003), 33(3), 150-152
 CODEN: RHGOE8; ISSN: 1001-1803
 PUBLISHER: Qingyongyebu Kexue Jishu Qingbao Yanjiusuo
 DOCUMENT TYPE: Journal
 LANGUAGE: Chinese

L20 ANSWER 9 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB The present invention generally relates to a process for the skeletal isomerization of unsatd. linear fatty acids and/or alkyl esters thereof to their branched counterparts with improved selectivity and conversion. Said skeletal isomerization process comprises contacting said unsatd. linear fatty acids and/or alkyl esters thereof with at least one metal ion-exchanged solid acid catalyst such as zeolite catalyst. The present invention also relates to a process for the preparation of branched fatty acids and/or alkyl esters thereof from their straight chain counterparts. Finally, the invention also relates to various derivs. prepared from the branched fatty acids and/or alkyl esters prepared in accordance with the present invention.

ACCESSION NUMBER: 2004:857220 CAPLUS

DOCUMENT NUMBER: 141:333929

TITLE: Metal ion-exchanged solid materials as catalysts for the skeletal isomerization of fatty acids and alkyl esters thereof

INVENTOR(S): Zhang, Zongchao; Zhang, Shuguang

PATENT ASSIGNEE(S): USA

SOURCE: U.S. Pat. Appl. Publ., 14 pp.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004204598	A1	20041014	US 2003-412201	20030411
WO 2004089854	A1	20041021	WO 2004-EP3068	20040323
WO 2004089854	C1	20050310		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, GU, HT, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2003-412201 A 20030411

L20 ANSWER 11 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB The composition comprises (a) a photopolymerizable urethane acrylate oligomer containing polydimethylsiloxane; (b) a monomer; (c) a photoinitiator; (d) a leveling/defoaming agent; and (e) an antioxidant. The photopolymerizable urethane acrylate oligomer containing polydimethylsiloxane is prepared (i) a first polyol compound containing polydimethylsiloxane, (ii) a second polyol compound having mol. weight 100-10,000, (iii) a polyisocyanate, (iv) an acrylate alc., (v) a urethane reaction catalyst, and (vi) a polymerization inhibitor. The optical loss after the ribbon manufacturing

process can be minimized by increasing tensile strength and surface slipping characteristics and by minimizing the shrinkage of resin when it is cured. Thus, 60 parts oligomer prepared from Hsi 2111(hydroxy-terminated polydimethylsiloxane), tetrahydrofuran-propylene oxide copolymer diol, polycaprolactone polyol, IPDI and 2-hydroxypropyl acrylate was mixed with isobornyl acrylate 15, N-vinylpyrrolidone 10, photoinitiator 8 and other additives 7 parts to give a optical fiber ribbon showing friction force 240 x 10-3 kg and tensile strength 83 kg/mm2.

ACCESSION NUMBER: 2004:618735 CAPLUS

DOCUMENT NUMBER: 141:157896

TITLE: Resin composition containing polydimethylsiloxane-containing urethane acrylic oligomer for manufacturing optical fiber ribbon

INVENTOR(S): Chang, Se-lee; Oh, Jung-hyun; Oh, Woo-jeong; Jung, Ki-sung

PATENT ASSIGNEE(S): SSCP Co., Ltd., S. Korea

SOURCE: U.S., 9 pp.

CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6770685	B1	20040803	US 2000-690271	20001017
KR 2001037679	A	20010515	KR 1999-45335	19991019
			KR 1999-45335	A 19991019

PRIORITY APPLN. INFO.: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 10 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB Synthesis and application are claimed for lubricants additives containing metal dialkyl dithiocarbamate and zinc dialkyl dithiophosphate. The latter is a product of reaction of phosphorus pentasulfide with C3-C8-alc. or C3-C12-alkylphenol followed by neutralization with zinc oxide. Metal dialkyl dithiocarbamate has general formula [R2NC(S)S]nMe, wherein Me is copper, nickel, cobalt, zinc, molybdenum, or cadmium; n valence of metal; and R C1-C8-alkyl radical. Lubrication composition contains 0.5 to 2.0 wt % of the

indicated additive. Motor oil additive compns. are also claimed that contain high-alkalinity sulfonate, phenate, alkali metal alkylsalicylate, ashless dispersant, and the additive above mentioned. The formulations claimed owe upgraded antioxidn., antivear, and metal-washing properties, they differ with reduced content of phosphorus and prolong lifetime of afterburner catalysts.

ACCESSION NUMBER: 2004:847562 CAPLUS

DOCUMENT NUMBER: 142:8956

TITLE: Metal dialkyl dithiocarbamate and zinc dialkyl dithiophosphate preparation and their use as fuel additives

PATENT ASSIGNEE(S): OOO "RESSELL GRUP", Russia

SOURCE: Russ., No pp. given

CODEN: RUKKE7

DOCUMENT TYPE: Patent

LANGUAGE: Russian

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
RU 2237705	C1	20041010	RU 2003-110861	20030416
PRIORITY APPLN. INFO.:			RU 2003-110861	20030416

L20 ANSWER 12 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB The invention is related to a process for synthesizing amines and polyamines, in particular isophorone diamine (IPDA), by hydrogenation of substrates or intermediates containing a nitrile and an imine group in the presence of a catalyst based on a Group VIII metal, water and ammonia in a condensed phase, and advantageously liquid phase. The process eliminates side reactions such as transamination and reduction of

the ketone to the alc. Dry Raney type Co catalyst was added to vials containing stock solns. of isophorone nitrile dissolved in methanol, H2O, water and an internal standard, and the reactor was pressurized with 35 bar H2 for 6 h to give IPDA in 55% yield and cis/trans ratio = 13.7. Similarly, addition of acetylacetonates of Hf, Cr, and Ir through liquid phase on the catalyst with low Ni content provided a decrease in the cis/trans ratio to about 3.3.

ACCESSION NUMBER: 2004:589534 CAPLUS

DOCUMENT NUMBER: 141:140103

TITLE: Process for synthesizing amines, in particular isophorone diamines, by hydrogenation of substrates and intermediates containing both a nitrile and an imine group in the presence of Raney type catalyst, especially Co-based, water and ammonia

INVENTOR(S): Marion, Philippe; Lowe, David Michael; Volpe, Anthony F., Jr.; Weiskamp, Thomas

PATENT ASSIGNEE(S): Rhodia Chimie, Fr.; Rhone Poulenc Chimie

SOURCE: PCT Int. Appl., 31 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004060866	A2	20040722	WO 2003-US41441	20031231
WO 2004060866	A3	20040923		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, GU, HT, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SV, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-437155P P 20021231

OTHER SOURCE(S): CASREACT 141:140103

L20 ANSWER 13 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Alkylation of ethylenediamine with alcs. using
 CuO-ZnO/Al₂O₃ catalysts prepared with various methods was
 investigated in the liquid phase. N-Methylethylenediamine was
 mainly obtained when methanol was used as alc., and its yield
 was largely dependent on the preparation method. XRD spectra of the
 catalysts revealed that Cu metal was the active species,
 and that a catalyst possessing smaller Cu metal
 particles showed the higher activity. Tert-Bu alc. did not
 give N-alkylation products, suggesting a reductive alkylation mechanism.
 As for the reactions with other polyamines and alcs., N-
 cyclohexylethylenediamine was efficiently formed with high
 selectivity.

ACCESSION NUMBER: 2004:384995 CAPLUS
 DOCUMENT NUMBER: 141:331775
 TITLE: Alkylation of ethylenediamine with alcohols
 by use of Cu-based catalysts in
 the liquid phase
 AUTHOR(S): Yamakawa, Tetsu; Tsuchiya, Ikuyo; Mitsuzuka, Daisuke;
 Ogawa, Tsukasa
 CORPORATE SOURCE: Sayami Chemical Research Center, Ayase-shi, Kanagawa,
 252-1193, Japan
 SOURCE: Catalysis Communications (2004), 5(6), 291-295
 CODEN: CCAOAC; ISSN: 1566-7367
 PUBLISHER: Elsevier Science B.V.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 14 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The two-component polyurethane binder system comprises: a first component
 containing at least one compound having active hydrogen atoms capable of
 reacting with isocyanate functionality, at least one catalyst to
 accelerate the reaction and formation of urethane linkages, and an
 effective amount of fumed silica to delay the action of the catalyst
 ; and a second component containing at least one polyisocyanate. Thus, a
 composition was prepared from A component containing polyether diol 25.05,
 polyether
 triol 3.78, dipropylene glycol 8.32, ricinus oil 18.90, barium sulfate
 15.12, calcium carbonate 19.66, iron oxide 2.45, fluorosurfactant 0.05,
 organotin catalyst 0.02, iron acetylacetonate 0.02, C7-11
 alcs. phthalate 3.78, mol. sieve 1.25, polysiloxane 0.30, hindered
 amine UV stabilizer 0.80, and fumed silica 0.50 wt% and B
 component containing modified diphenylmethane 4,4'-diisocyanate 20.00, high
 2,4 isomer blend of diphenylmethane diisocyanate 26.00, and polyether diol
 54.00 wt%. The resulting polyurethane surface exhibited a gel time of 18
 min and a sufficient cure time for an athletic surface application.

ACCESSION NUMBER: 2004:352993 CAPLUS
 DOCUMENT NUMBER: 140:358559
 TITLE: Two-component polyurethane binders for athletic
 surface
 INVENTOR(S): Croley, Tim
 PATENT ASSIGNEE(S): USA
 SOURCE: U.S. Pat. Appl. Publ., 9 pp.
 CODEN: USXXCO
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004081830	A1	20040429	US 2002-282591	20021029
WO 2004039889	A1	20040513	WO 2003-US34066	20031027
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
PRIORITY APPLN. INFO.:			US 2002-282591	A 20021029

L20 ANSWER 15 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB A novel multinucleating chiral reaction field is designed by the intensive
 assembling of characteristically functionalized metals which play specific
 roles in controlling the stereochem. course. Under the developed chiral
 circumstances highly efficient asym. reactions were developed which could
 not be realized in the ever developed monometallic reaction field.
 Enantioselective 1,3-dipolar cycloaddn. reaction of nitrile oxides and a
 nitrene to allylic alcs. was achieved by using zinc
 and magnesium metal and diisopropyl (R,R)-tartrate as a chiral auxiliary
 to afford the corresponding 2-isoxazolines and isoxazolidines with
 excellent enantioselectivity. The first enantioselective hetero
 Diels-Alder reaction of a nitroso compound was realized to afford the
 corresponding dihydro-1,2-oxazine with excellent enantioselectivity. The
 Asym. Nucleophilic addition to imines were also achieved. Optically active
 γ-lactams can be synthesized from the reaction of N-tosyl
 homoallylamines catalyzed by palladium and copper salts under CO
 and O in the presence of chiral ligand.

ACCESSION NUMBER: 2004:306484 CAPLUS
 DOCUMENT NUMBER: 141:206551
 TITLE: Development of highly efficient asymmetric reactions
 based on the construction of functionalized
 multinucleating chiral reaction field
 AUTHOR(S): Ukaji, Yutaka
 CORPORATE SOURCE: Department of Chemical Science, Graduate School of
 Natural Science and Technology, Kanazawa University,
 Japan
 SOURCE: Asahi Garasu Zaidan Josei Kenkyu Seika Hokoku (2003)
 No pp. given
 CODEN: AGSHEN; ISSN: 0919-9179
 URL: <http://www.af-info.or.jp/jpn/subsidy/report2/2003/body/03A-C08-P069.TXT>
 PUBLISHER: Asahi Garasu Zaidan
 DOCUMENT TYPE: Journal; (online computer file)
 LANGUAGE: Japanese

L20 ANSWER 16 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Transition metal-based Lewis acids, such as SnCl₄·5H₂O,
 Co(OAc)₂·4H₂O, Ni(OAc)₂·2H₂O, NiCl₂, Mn(OAc)₂·4H₂O,
 etc., catalyze the nucleophilic opening of epoxide rings by amines
 leading to the efficient synthesis of β-amino alcs. The
 reaction works well with aromatic and aliphatic amines in high yields under
 solvent-free conditions.

ACCESSION NUMBER: 2004:302824 CAPLUS
 DOCUMENT NUMBER: 141:6630
 TITLE: Transition metal-based Lewis acid-catalyzed ring
 opening of epoxides using amines under solvent-free
 conditions
 AUTHOR(S): Zhao, Pei-Qing; Xu, Li-Wen; Xia, Chun-Gu
 CORPORATE SOURCE: State key laboratory of Oxo Synthesis and Selective
 Oxidation, Lanzhou Institute of Chemical Physics,
 Chinese Academy of Sciences, Lanzhou, 730000, Peop.
 Rep. China
 SOURCE: Synlett (2004), (5), 846-850
 CODEN: SYNLES; ISSN: 0936-5214
 PUBLISHER: Georg Thieme Verlag
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 141:6630
 REFERENCE COUNT: 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 17 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A method of forming a plurality of monodisperse nanoparticles. Each of the nanoparticles comprises a nanocryst. inorg. core and at least one outer coating comprising an ionizable stabilizing material that substantially covers the core. The method comprises the steps of: combining a nonpolar aprotic organic solvent, an oxidant, and a 1st surfactant; providing at least one organometallic compound to the combined nonpolar aprotic organic solvent, oxidant, and 1st surfactant; and heating the combined nonpolar aprotic organic solvent, oxidant, 1st surfactant, and the at least one organometallic compound under an inert gas atmospheric to a 1st temperature in a range from .apprx.30° to .apprx.400° for a 1st time interval, thereby reacting at least one organometallic compound and the oxidant in the presence of the 1st surfactant and the nonpolar aprotic organic solvent to form a plurality of nanoparticles, each of the plurality of nanoparticles comprising a nanocryst. inorg. core and at least one outer coating comprising the 1st surfactant. At least one organometallic compound comprises a metal and at least one ligand.

ACCESSION NUMBER: 2004:100564 CAPLUS

DOCUMENT NUMBER: 140:148452

TITLE: Method of making crystalline nanoparticles from organometallic and transition metal complexes

INVENTOR(S): Bonitatebus, Peter John; Acar, Havva Yagci

PATENT ASSIGNEE(S): General Electric Company, USA

SOURCE: U.S. Pat. Appl. Publ., 16 pp.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004022937	A1	20040205	US 2002-208945	20020731
JP 2004067508	A2	20040304	JP 2003-282299	20030730
CN 1475460	A	20040218	CN 2003-152227	20030731
EP 1394223	A1	20040303	EP 2003-254809	20030731

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK

PRIORITY APPLN. INFO.: US 2002-208945 A 20020731

L20 ANSWER 18 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A review. The use of copper and related complexes in applications to organic synthesis is reviewed.

ACCESSION NUMBER: 2003:1000504 CAPLUS

DOCUMENT NUMBER: 141:242819

TITLE: Product class 4: organometallic complexes of copper

AUTHOR(S): Heaney, H.; Christie, S.

CORPORATE SOURCE: Dept. of Chemistry, University of Loughborough, Loughborough, LE11 3TU, UK

SOURCE: Science of Synthesis (2004), 3, 305-662

CODEN: SSCYJ9

PUBLISHER: Georg Thieme Verlag

DOCUMENT TYPE: Journal; General Review

LANGUAGE: English

REFERENCE COUNT: 1706 THERE ARE 1706 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 19 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB The invention is directed to a process of arylation, vinylation or alkynylation of nucleophilic compds., in particular to arylation of nitrogen-containing nucleophiles. The process of arylation, vinylation or alkynylation involves reaction of a nucleophilic compound with a compound carrying a leaving group in the presence of an effective quantity of a catalyst based on a metal element M chosen from groups VIII, IB and IIB of the periodic table and of a bidentate, tridentate or tetradentate ligand with at least one imino group and at least an addl. nitrogen atom as chelation sites. The advantages include moderate reaction temperature, lower reaction time, use of aryl bromides and chlorides beside iodides as arylation agents, and of the more economic Cu catalyst. Preferred nucleophiles include nitrogen heterocycles such as imidazole, pyrazole, pyrazine, sulfonamides, thio/ols., and malonates. The operating protocol comprises successive introduction of the following to a 35 mL Schlenk tube under nitrogen: 0.05 mmol Cu catalyst, 0.1 mmol ligand, 0.75 mmol nucleophile, 1 mmol base, 0.5 mmol iodobenzene, and 300 mmol MeCN, followed by agitation and heating of the tube and its contents in an oil bath. For example, 80% 1-phenyl-1H-pyrazole was obtained from pyrazole and bromobenzene using Cu2O and trans-N,N'-bis(2-thiophenemethylene)-1,2-cyclohexanediamine in MeCN at 82° for 24 h.

ACCESSION NUMBER: 2003:950190 CAPLUS

DOCUMENT NUMBER: 140:16744

TITLE: Process for arylation, vinylation or alkynylation of nucleophilic compounds, in particular

nitrogen-containing nucleophiles

INVENTOR(S): Taillefer, Marc; Cristau, Henri Jean; Cellier, Pascal;

Spindler, Jean Francis

PATENT ASSIGNEE(S): Rhodia Chimie, Fr.

SOURCE: Fr. Demande, 91 pp.

CODEN: FRCKBL

DOCUMENT TYPE: Patent

LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2840303	A1	20031205	FR 2002-6717	20020531
WO 2003101966	A1	20031211	WO 2003-FR1647	20030602

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MN, MW, MX, MY, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SH, TD, TG

EP 1509502 A1 20050302 EP 2003-756038 20030602

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK

PRIORITY APPLN. INFO.: FR 2002-6717 A 20020531
WO 2003-FR1647 W 20030602

OTHER SOURCE(S): CASREACT 140:16744; MARPAT 140:16744

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 19 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

L20 ANSWER 20 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB The moldings containing 21 metals chosen from Group VIII transition metals, Mn, Cu, and Zn, useful for packaging, are stored in packaging materials and/or containers comprising oxygen-barrier materials and compns. having oxygen-scavenging rate 22 times that of the polyamide moldings. During the storage, the sum of (A) initial O amount in the containers and (B) the product of storage days and O permeation/day from exterior to the packaging materials and/or containers is 80% the O-scavenging property of the compns. Thus, a multilayer film comprising polyethylene/adhesive/adipic acid-m-xylylenediamine copolymer (containing Co stearate)/adhesive/polyethylene was stored in a an Al-laminated film with a CaCl₂/Fe mixture (oxygen-scavenging property 6250 mL) sealed in a porous polyethylene bag at room temperature for 6 mo to show both initial and final O-scavenging property 120 mL.

ACCESSION NUMBER: 2003:918617 CAPLUS
DOCUMENT NUMBER: 139:382507
TITLE: Long-term storage of oxygen-scavenging polyamide moldings
INVENTOR(S): Otaki, Ryoji
PATENT ASSIGNEE(S): Mitsubishi Gas Chemical Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKKKAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003335379	A2	20031125	JP 2002-141432	20020516

PRIORITY APPLN. INFO.: JP 2002-141432 20020516

L20 ANSWER 21 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB Title compds. are prepared by reaction of R1CN (R1 = H, aliphatic, aromatic, or araliph. group) with H₂NCHR₂CH₂NHR₄ (R2, R3 = H, aliphatic, aromatic, or araliph. group; R4 = aliphatic, aromatic, or araliph. group) in the presence of catalysts chosen from metal oxides, metal salts, organic acids, or solid acids and dehydrogenation under heating using Ni, Pd, Pt, and/or Cu catalysts. Quaternary imidazolium salts are prepared by quaternization of the imidazoles. N-ethylethylenediamine was cyclocondensed with MeCN in MeOH in the presence of Zn acetate at 200° for 3 h to give 1-ethyl-2-methylimidazoline with 93% selectivity at 93% conversion, which was heated with N 103B at 180° for 5 h to give 1-ethyl-2-methylimidazole with 92% selectivity at 90% conversion.

ACCESSION NUMBER: 2003:870612 CAPLUS
DOCUMENT NUMBER: 139:350738
TITLE: Preparation of N-substituted imidazoles
INVENTOR(S): Takahashi, Fumiharu; Yoshimura, Hiroyuki
PATENT ASSIGNEE(S): Tosoh Corp., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.
CODEN: JKKKAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003313172	A2	20031106	JP 2002-120563	20020423

PRIORITY APPLN. INFO.: JP 2002-120563 20020423
OTHER SOURCE(S): CASREACT 139:350738; MARPAT 139:350738

L20 ANSWER 22 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB Catalytic compns. are suited for use in hydrogenation processes for organic compds., as in amination of alcs. or hydrogenation of nitro groups to the amine. The catalytic composition is an improvement in Ni catalysts promoted with Pd carried on a support. The improvement resides in including a promoting effect metal M and/or its oxide, selected from Zn, Cd, Cu, and Ag, typically .apprx.0.01-10% (based on weight of the support). The presence of Zn in the Pd/Ni monolith catalyst effectively suppressed formation of lights and tars (by products) even at concns. as low as 0.3 wt% based on support.

ACCESSION NUMBER: 2003:870476 CAPLUS
DOCUMENT NUMBER: 139:339277
TITLE: Metal modified Pd/Ni catalysts and hydrogenation
INVENTOR(S): Ding, Hao
PATENT ASSIGNEE(S): Air Products and Chemicals, Inc., USA
SOURCE: Eur. Pat. Appl., 9 pp.
CODEN: EPXKDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1358935	A1	20031105	EP 2003-8826	20030424

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK
US 2003207761 A1 20031106 US 2002-137053 20020501
US 6762324 B2 20040713
BR 2003001141 A 20040817 BR 2003-1141 20030428
CN 1454713 A 20031112 CN 2003-128451 20030429
JP 2003340283 A2 20031202 JP 2003-124631 20030430
US 2004199020 A1 20041007 US 2004-824993 20040415
US 2004199017 A1 20041007 US 2004-825027 20040415
US 2005038295 A1 20050217 US 2004-824952 20040415
US 6878849 B2 20050412
PRIORITY APPLN. INFO.: US 2002-137053 A 20020501
REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 23 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB The present invention relates to a process for the epoxidn. of 21 organic compound with an oxygen-delivering substance, for example a hydroperoxide, in the presence of 21 catalyst containing a metal-organic framework material comprising pores and a metal ion and 21 bidentate organic compound, said bidentate organic compound being coordinately bound to the metal ion. Thus, a 66:24:10 volume ratio of O₂, He, and propylene was streamed through a tube reactor containing AgNO₃-treated HOF-5 at 220° to give propylene oxide with a turnover of 3.3% and selectivity of 10.3% after 15 h.

ACCESSION NUMBER: 2003:747904 CAPLUS
DOCUMENT NUMBER: 139:278233
TITLE: Process for epoxidation of organic compounds with oxygen or oxygen-delivering compounds using catalysts containing metal-organic framework (MOF) materials
INVENTOR(S): Mueller, Ulrich; Lobrea, Lisa; Hesse, Michael; Yaghi, Omar M.; Eddaoudi, Mohamed
PATENT ASSIGNEE(S): BASF Aktiengesellschaft, Germany; The Regents of the University of Michigan
SOURCE: U.S., 13 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6624318	B1	20030923	US 2002-157494	20020530

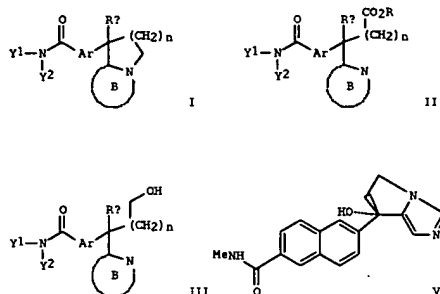
WO 2003101975 A1 20031211 WO 2003-EP5547 20030527
W: US
RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR
EP 1513823 A1 20050316 EP 2003-730125 20030527
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, FI, RO, CY, TR, BG, CZ, EE, HU, SK
PRIORITY APPLN. INFO.: US 2002-157494 A 20020530
WO 2003-EP5547 W 20030527

OTHER SOURCE(S): CASREACT 139:278233
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 24 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Gel composition of TiO₂ precursor is produced by heating a Ti alkoxide mixed solution at 100-200°. The mixed solution comprises 1 mol of Ti alkoxide, 40-120 mol of alkylene glycol, 1.0-20 mol of amino alc. (dialkanolamine and/or trialkanolamine), 10-80 mol of H₂O, and 50.1 mol of metal or metal ion selected from 21 of Cu, Cu₂, Mn, Mn₂, Ni, Ni₂, Co, Co₂, Zn, and Zn₂. TiO₂ is produced by calcining the precursor at 500-650° under inert gas atmosphere. The TiO₂ thin film has a photocatalytic characteristics, and is useful for screen printing.
 ACCESSION NUMBER: 2003:706872 CAPLUS
 DOCUMENT NUMBER: 139:232539
 TITLE: Method for producing titanium dioxide and its precursor
 INVENTOR(S): Nishizawa, Hitoshi
 PATENT ASSIGNEE(S): Japan Science and Technology Corporation, Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 14 pp.
 CODEN: JI00KAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003252626	A2	20030910	JP 2002-57259	20020304
PRIORITY APPLN. INFO.:			JP 2002-57259	20020304

L20 ANSWER 25 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 GI



AB Disclosed are a process for industrially advantageously producing a steroid C17,20-lyase inhibitor represented by the following general formula [I]: Ra = H, a substituent; Ar = (un)substituted aromatic hydrocarbyl;
 Y1, Y2 = H, a substituent; the ring B = (un)substituted N-containing ring;
 n = an integer of 1-3 and a Reformatskii reagent in a stable form which is suitable for use in the production process. Either a specific β -hydroxy ester compound derivative (II; R = an ester residue; Ra, Ar, the ring B, Y1, Y2, n = same as above) obtained from a specific carbonyl compound by the Reformatskii reaction or a salt of the compound is reduced in the presence of a metal/hydrogen complex compound and a metal halide to an alc. (III; Ra, Ar, the ring B, Y1, Y2, n = same as above) and then subjected to ring closure to thereby obtain a compound represented by the general formula I. In the Reformatskii reaction, a stable solution of the compound represented by BrZnCH₂CO₂CH₃ or crystals of the compound represented by (BrZnCH₂CO₂Et.THF)₂ are useful. Thus, 10 L THF and 253 mL chlorotriethylsilane were successively added to 2,616 g in powder, stirred at 25° for 30 min, treated dropwise with a solution of 2,212 mL Et bromoacetate in 25 L THF, and stirred at 31-35° for 30 min to give a Reformatskii reagent solution which was treated with 21.2 g (+)-cinchonine at 0-5° and then dropwise with 18.6 mL pyridine at 0-5° over 7 min, stirred at 0-5° for 20 min, treated dropwise with a solution of 30 g N-methyl-6-[(1-trityl-1H-imidazol-4-yl)carbonyl]-2-naphthamide in 300 mL THF over 30 min at -42° to -40°, and stirred at -45° to -40° for 1 h to give, after workup, 29.2 g Et (3S)-3-hydroxy-3-[6-[(methylamino)carbonyl]-2-naphthyl]-3-[(1-trityl-1H-imidazol-4-yl)propanoate (IV) (83% yield, 93.5% ee). THF (13 mL) and 0.645 g NaBH₄ were successively added to 1.3 g IV and the resulting mixture was treated with 0.95 g CaCl₂ at 2° and

L20 ANSWER 25 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 then dropwise with 13 mL ethanol over 15 min at 2°, stirred at 3-4° for 30 min and at 40-43° for 4 h to give, after workup, 1.08 g 6-[(1S)-1,3-dihydroxy-1-[(1-trityl-1H-imidazol-4-yl)propyl]-N-methyl-2-naphthamide (V) (89% yield, 92.0% ee). THF (7 mL) and 0.42 mL diisopropylethylamine were successively added to 0.35 g V and the resulting mixt. was treated dropwise with 0.07 mL methanesulfonyl chloride at 0-5°, stirred at 0-5° for 40 min, treated with 1.8 mL MeOH and 3.5 mL MeCN, and stirred at 60-65° for 4 h to give, after workup, 0.87 g 6-[(1S)-7-hydroxy-6,7-dihydro-5H-pyrrolo[1,2-c]imidazol-7-yl]-N-methyl-2-naphthamide (VI) (62%, 98.2% ee).
 ACCESSION NUMBER: 2003:570964 CAPLUS
 DOCUMENT NUMBER: 139:133566
 TITLE: Process for producing fused imidazole compound, Reformatskii reagent in stable form, and process for producing the same
 INVENTOR(S): Kawakami, Jun-ichi; Nakamoto, Koji; Nuwa, Shigeru; Handa, Syoji; Miki, Shokyo
 PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan
 SOURCE: PCT Int. Appl., 141 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003059889	A1	20030724	WO 2003-JP92	20030109
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LX, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, VU, ZA, ZH, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
JP 2004161726	A2	20040610	JP 2003-3231	20030109
EP 1471056	A1	20041027	EP 2003-700504	20030109
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
US 2005043544	A1	20050224	US 2004-500999	20041001
PRIORITY APPLN. INFO.:			JP 2002-3821	A 20021011
			JP 2002-279438	A 20020925
			WO 2003-JP92	W 20030109
OTHER SOURCE(S):		MARPAT 139:133566		
REFERENCE COUNT:		5	THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT	

L20 ANSWER 26 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Reactive oxygen-derived species and particularly OH radicals can degrade hyaluronic acid (HA), resulting in a loss of viscosity and a subsequent decrease in its effectiveness as a joint-lubricating agent. The production of OH in the vicinity of HA can be catalyzed by bound redox-active metals, which participate in the Haber-Weiss reaction. Damage to HA can also occur as a result of hypochlorite formed by myeloperoxidase (MPO). The protective reagents commonly used to inhibit oxidative stress-induced degradation of HA include antioxidative enzymes, such as SOD and catalase, chelators that coordinate metal ions rendering them redox-inactive, and scavengers of radicals, such as OH, as well as nonradical reactive species. In recent years, stable cyclic nitroxides have also been widely used as effective antioxidants. In many cases, nitroxide antioxidants operate catalytically and mediate their protective effect through an exchange between their oxidized and reduced forms. It was anticipated, therefore, that nitroxides would protect HA from oxidative degradation as well. On the other hand, nitroxides serve as catalysts in many oxidation reactions of alcs., sugars and polysaccharides, including hyaluronan. Such opposite effects of nitroxides on oxidative degradation are particularly intriguing and the aim of the present study was to examine their effect on HA when subjected to diverse forms of oxidative stress. The results indicate that nitroxides protect HA from OH radicals generated enzymically or radiolytically. The protective effect is attributable neither to the scavenging of OH nor to the oxidation of reduced metal, but to the reaction of nitroxides with secondary carbohydrate radicals-most likely peroxyl radicals.
 ACCESSION NUMBER: 2003:521567 CAPLUS
 DOCUMENT NUMBER: 139:391295
 TITLE: Do stable nitroxide radicals catalyze or inhibit the degradation of hyaluronic acid?
 AUTHOR(S): Lurie, Ziva; Offer, Tal; Russo, Angelor; Samuni, Amram; Nitzan, Dorrit
 CORPORATE SOURCE: Department of Molecular Biology, Hebrew University-Hadassah Medical School, Jerusalem, Israel
 SOURCE: Free Radical Biology & Medicine (2003), 35(2), 169-178
 CODEN: FREMEH; ISSN: 0891-5849
 PUBLISHER: Elsevier Science Inc.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 REFERENCE COUNT: 38
 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 27 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The present invention relates to a process and apparatus for processing agricultural waste to make alc. and/or biodiesel. The agricultural wastes are subjected to anaerobic digestion which produces a biogas stream containing methane, which is subsequently reformed to a syngas containing carbon monoxide and hydrogen. The syngas is converted to an alc. which may be stored, sold, used, or fed directly to a reactor for production of biodiesel. The solids effluent from the anaerobic digester can be further utilized as slow release, organic certified fertilizer. Addnl., the wastewater from the process is acceptable for immediate reuse in agricultural operations.

ACCESSION NUMBER: 2003:472849 CAPLUS
 DOCUMENT NUMBER: 139:24090
 TITLE: System and method for extracting energy from agricultural waste
 INVENTOR(S): Branson, Jerrel Dale
 PATENT ASSIGNEE(S): Best Biofuels, LLC C/O Smithfield Foods, Inc., USA
 SOURCE: U.S. Pat. Appl. Publ., 16 pp.
 CODEN: USXXCO
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2003111410	A1	20030619	US 2002-320744	20021217
US 6824682	B2	20041130		
WO 2003051803	A1	20030626	WO 2002-US40116	20021217

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

EP 1456157 A1 20040915 EP 2002-790135 20021217
 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK

BR 2002015051 A 20041207 BR 2002-15051 20021217
 PRIORITY APPLN. INFO.: US 2001-340491P P 20011218
 WO 2002-US40116 W 20021217

L20 ANSWER 28 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Aluminophosphate (ALPO) and silicoaluminophosphate (SAPO) mol. sieves are prepared by: (1) forming a reaction mixture consisting of an alumina source, a phosphate source, optionally a silica source, and a nitrogen-containing (amine) template, (2) inducing crystallization to form a slurry, and (3) recovering the desired catalyst. The pH of the reactant precursor slurry is reduced after crystallization, and the slurry is held at <100° (preferably at or below ambient temperature) following crystallization. The method can be used to prepare such materials as SAPO-17, SAPO-18, SAPO-34, SAPO-35, SAPO-44, SAPO-47, ALPO-5, ALPO-11, ALPO-18, ALPO-34, ALPO-36, ALPO-37, and ALPO-46, and includes metal-containing forms of these materials, by using a metal oxide as the metal precursor (M = Zn, Mg, Mn, Co, Ni, Ga, Fe, Ti, Zr, Ge, Sn, Cr, and Cu). Such catalysts are useful for conversion of oxygenates (especially alcs.) to olefins.

ACCESSION NUMBER: 2003:319434 CAPLUS
 DOCUMENT NUMBER: 138:323744
 TITLE: Preparation of aluminophosphate and silicoaluminophosphate molecular sieves as conversion catalysts for oxygen-containing compounds to olefins
 INVENTOR(S): Mertens, Machteld Maria; Engels, Brita
 PATENT ASSIGNEE(S): Belg.
 SOURCE: U.S. Pat. Appl. Publ., 9 pp.
 CODEN: USXXCO
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2003078159	A1	20030424	US 2001-2241	20011023
WO 2003035549	A1	20030501	WO 2002-US23525	20020724

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZM, ZW

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2001-2241 A 20011023

L20 ANSWER 29 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB High activity, supported, nanosized metallic catalysts for methanol reformation and methods of fabricating such catalysts are disclosed. In one embodiment, soluble metal species are dissolved in a polyhydroxylic alc. (polyol) solution. Platinum and ruthenium are preferred metal species. Other soluble metal species can be used, such as soluble Group 6, 7 and 8 metals. The polyol solvent is preferably a viscous alc., such as a diol, triol, or tetrol, to minimize particle diffusion and inhibit particle growth. The polyol solution is heated to reduce the metal(s) to a zero valent state. Typically, the heating temperature will range from 20° to 300°, and the heating period will range from 1 min to 5 h. A high surface area conductive support material can be mixed with the polyol solution to form the supported catalysts in situ. Activated carbon, metals, and metal oxides, having a surface area from 20 to 2000 m²/g, are typical support materials.

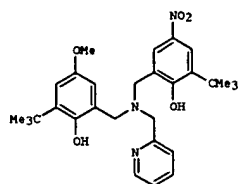
ACCESSION NUMBER: 2003:312665 CAPLUS
 DOCUMENT NUMBER: 138:306834
 TITLE: Preparation of supported nano-sized catalyst particles via a polyol process for methanol reforming
 INVENTOR(S): Laine, Richard M.; Sellinger, Alan
 PATENT ASSIGNEE(S): Canon Kabushiki Kaisha, Japan
 SOURCE: U.S., 19 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6551960	B1	20030422	US 2000-596764	20000619
			US 2000-596764	20000619

PRIORITY APPLN. INFO.: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 30 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Unsym. substituted conjugated diynes are prepared selectively from alkynes and 1,1-dichloroethene by Pd-catalyzed reaction of terminal alkynes RC.tpbond.CH (R = BuCH₂, Ph, Me₃Si) with 1,1-dichloroethene, elimination of chloride and lithiation of the intermediate vinyl chlorides with 2 equiv of lithium diisopropylamide, transmetalation with either 2nBr₂ or 2nCl₂, and Pd-catalyzed cross-coupling of the alkadiynylzinc reagents with aryl and alkenyl iodides and bromides. Terminal alkynes are coupled by treatment of terminal alkynes with 1,1-dichloroethene in the presence of tetrakis(triphenylphosphine)palladium and copper (I) iodide to give the chlorovinylated alkynes as the major products in 66-82% yields with 0-5% of the 1,1-bis(alkynyl)ethenes as byproducts; an alternative route uses the coupling of an octynylzinc reagent (generated from 1-octyne by lithiation followed by transmetalation) with 1,1-dichloroethene in the presence of bis(tri-tert-butylphosphine) to give a chlorovinylalkyne in 33% yield with 25% yield of the bis(octynyl)ethene byproduct. Treatment of (chlorovinyl)alkynes with LDA generated from diisopropylamine and butyllithium followed by the addition of zinc bromide gives an alkadiynylzinc reagent which undergoes stereoselective coupling reactions with aryl and vinyl iodides and bromides such as iodobenzene, trans-1-iodo-1-octene, Me (E)-3-bromo-2-methylpropenoate, 2-iodothiophene, and trans-(β-bromovinyl)trimethylsilylacetylene to give the desired unsym. diynes in 67-92% yields. Trans-3-iodo-2-propen-1-ol is also an effective coupling partner for the alkadiynylzinc reagents if diethylzinc is added to the iodoallylic alc. before addition to the alkadiynylzinc reagent. The use of 1,1-dichloroethene (\$17.50/100g) renders this method more economical than those involving 1,2-dihaloethylenes such as 1,2-dichloroethene (\$151.80/100g) reported previously.

ACCESSION NUMBER: 2003:263923 CAPLUS
 DOCUMENT NUMBER: 139:6583
 TITLE: Strictly "Fair"-Selective and Economical Synthesis of Conjugated Diynes via Pd-Catalyzed Reaction of Terminal Alkynes with 1,1-Dichloroethene, Elimination with LDA, and Subsequent Transformations
 AUTHOR(S): Qian, Mingxing; Weyshi, Kiichi
 CORPORATE SOURCE: Herbert C. Brown Laboratories of Chemistry, Purdue University, West Lafayette, IN, 47907-2084, USA
 SOURCE: Organic Process Research & Development (2003), 7(3), 412-417
 CODEN: OPRDFK; ISSN: 1083-6160
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 139:6583
 REFERENCE COUNT: 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT



I

AB [Cu(LH)(OAc)], [Cu2(L)2] and [Zn(LH)(OAc)] (LH2 = I) were prepared and the crystal structure of [Cu(LH)(OAc)]·1/2CH2Cl2 was determined. The axial phenol group is protonated and the equatorial phenoxyl group antiferromagnetically coupled to the cupric center in [Cu(LH)(OAc)]⁺, which can be considered as both a structural and functional model of galactose oxidase. This complex reproduces the features of the enzyme chemical. The phenoxyl radical position (equatorial vs. axial) is dictated by protonation.

ACCESSION NUMBER: 2002:670868 CAPLUS
DOCUMENT NUMBER: 137:392518
TITLE: A structural and functional model of galactose oxidase: Control of the one-electron oxidized active form through two differentiated phenolic arms in a tripodal ligand
AUTHOR(S): Thomas, Fabrice; Gellon, Gisele; Gautier-Luneau, Isabelle; Saint-Aman, Eric; Pierre, Jean-Louis
CORPORATE SOURCE: Laboratoire de Chimie Biomimétique LECS, UMR CNRS 5616 Université J. Fourier, Grenoble, 38041/9, Fr.
SOURCE: Angewandte Chemie, International Edition (2002), 41(16), 3047-3050
CODEN: ACIEF5; ISSN: 1433-7851
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 137:392518
REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 33 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Amination of fatty alcs. by hydrogenation and dehydrogenation catalysis is one of the main com. processes for the production of dimethylalkylamines. The key factor in this process is the preparation of catalysts with high selectivity. A study of amination catalyzed by Cu-Ni catalysts supported on CaCO3 is reported in this paper. Selectivity of the catalysts was adjustable by varying the ratio of Cu to Ni or by adding a third element (Zn or Mg). The promotion of catalyst selectivity was mainly attributable to the effect of the components on the reducibility of Ni2+ in the catalysts. Lower reducibility of Ni2+ led to higher catalyst selectivity.

ACCESSION NUMBER: 2002:596283 CAPLUS
DOCUMENT NUMBER: 137:312671
TITLE: Study of the amination of fatty alcohols catalyzed by Cu-Ni catalysts
AUTHOR(S): Li, Qinxiao; Zhang, Gaoyong; Peng, Shaoyi
CORPORATE SOURCE: China Research Institute of Daily Chemical Industry, Taiyuan, 030001, Peop. Rep. China
SOURCE: Journal of Surfactants and Detergents (2002), 5(3), 229-233
CODEN: JSDEFL; ISSN: 1097-3958
PUBLISHER: AOC Press
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 32 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Four new long chain alkyl substituted 2,6-dioxo-1,4,7,10-tetrazacyclododecanes bearing an alc. pendant has been synthesized. They coordinate with metal ion (Zn2+, Cu2+, Ni2+, Co2+) to yield 1:1 five-coordinate complexes. The catalytic properties of these complexes have been investigated for the hydrolysis of bis(p-nitrophenyl) phosphate (BNPP) in comicellar solution. The alc. pendant has a profound effect on the catalytic properties of macrocyclic complexes, and the nature of transition metal ion, micellar microenvironment, hydrolysis temperature, hydrophobic interactions between the metalocatalyst and substrate are also important factors in the hydrolysis of BNPP. Under the physiol. conditions (pH=7.41, 35±0.1°), the complexes exhibit higher catalytic activity (up to over 2-3 orders of magnitude) than metal ion or ligand alone in the hydrolysis of BNPP in comicellar solution with 2 mM Brij 35.

ACCESSION NUMBER: 2002:641200 CAPLUS
DOCUMENT NUMBER: 138:102811
TITLE: Phosphodiester hydrolysis by metal ion macrocyclic dioxotetraamine complexes bearing alcohol pendant in comicellar solution
AUTHOR(S): Xiang, Qing-Xiang; Yu, Xiao-Qi; Su, Xiao-Yu; Yan, Qian-Shun; Wang, Tao; You, Jing-Song; Xie, Ru-Gang
CORPORATE SOURCE: Department of Chemistry, Sichuan University, Chengdu, 610064, Peop. Rep. China
SOURCE: Journal of Molecular Catalysis A: Chemical (2002), 187(2), 195-200
CODEN: JMCCF2; ISSN: 1381-1169
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:102811
REFERENCE COUNT: 36 THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 34 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Optically active amines R1R2CHNH3 (I; R1-R3 = (un)substituted (cyclo)alkyl, aryl(alkyl), heteroaryl, heterocyclyl; R3 = H; R1 = R2) were racemized by heating in the presence of H and a supported (de)hydrogenation catalyst containing Cu and ZnO as active components. The racemization of I can be carried out in the presence of secondary alcs. R1R2CHOH and/or asym. ketones R1COR2 (R1, R2 as defined for I). For example, passing 200 mL/h of 95.4% pure (S)-1-methoxy-2-propylamine [(S)-II] at 230° and 16 bar H (containing .apprx.20 volume% NH3) through a tubular reactor packed with catalyst containing CuO 66, ZnO 24, Al2O3 5 and Cu 5% (preparation given) gave a racemic mixture containing 51.9% (S)-II and 48.1% (R)-II.

ACCESSION NUMBER: 2002:463997 CAPLUS
DOCUMENT NUMBER: 137:48866
TITLE: Racemization of optically active amines
INVENTOR(S): Funke, Frank; Liang, Shelia; Kramer, Andreas; Stuermer, Rainer; Hoehn, Arthur
PATENT ASSIGNEE(S): Basf Aktiengesellschaft, Germany
SOURCE: Eur. Pat. Appl., 14 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1215197	A2	20020619	EP 2001-128602	20011130
EP 1215197	A3	20031029		
EP 1215197	B1	20050223		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IS, SI, LT, LV, FI, RO, HK, CY, AL, TR				
DE 10062729	A1	20020620	DE 2000-10062729	20001215
AT 289582	E	20050315	AT 2001-128602	20011130
US 2002120166	A1	20020829	US 2001-12344	20011212
US 6548704	B2	20030415		
CN 1363549	A	20020814	CN 2001-142892	20011214
JP 2002226437	A2	20020814	JP 2001-383504	20011217
US 6576795	B1	20030610	US 2002-261123	20021001
PRIORITY APPLN. INFO.:				
			DE 2000-10062729	A 20001215
			US 2001-12344	A3 20011212
OTHER SOURCE(S): MARPAT 137:48866				

L20 ANSWER 35 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Poly- or mono(hydroxymethyl)alkanals: R2C(CH2OH)CHO [R = C1-22
 (un)substituted aliphatic, aryl, arylalkyl, CH2OH; e.g., dimethylbutanal]
 are catalytically hydrogenated into their corresponding polyhydric
 alcs. (e.g., trimethylol) in the presence of a copper
 -containing catalyst (e.g., Raney copper) and the batch to
 be hydrogenated has a total content of 35 ppm of metal ions of the
 groups 3-14 of the periodic system and the ions are removed from the
 polyhydric alc. product by absorption, ion exchange, or
 chelation.

ACCESSION NUMBER: 2002:368435 CAPLUS
 DOCUMENT NUMBER: 136:368676
 TITLE: Method for the hydrogenation of poly- or
 mono(hydroxymethyl)alkanals into polyhydric alcohols
 using copper catalysts
 Dornbach, Matthias; Koch, Michael; Schulz, Gerhard;
 Weigl, Hagen; Maas, Steffen
 INVENTOR(S): BASF Aktiengesellschaft, Germany
 PATENT ASSIGNEE(S): PCT Int. Appl., 16 pp.
 SOURCE: CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002038525	A2	20020516	WO 2001-EP12681	20011102
WO 2002038525	A3	20020801		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
DE 10055180	A1	20020529	DE 2000-10055180	20001108
AU 2002016990	A5	20020521	AU 2002-16990	20011102
EP 1335891	A2	20030820	EP 2001-993593	20011102
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
BR 2001015199	A	20040217	BR 2001-15199	20011102
JP 2004513156	T2	20040430	JP 2002-541064	20011102
US 2004044256	A1	20040304	US 2003-399502	20030417
PRIORITY APPLN. INFO.: DE 2000-10055180 A 20001108 WO 2001-EP12681 W 20011102				
OTHER SOURCE(S): MARPAT 136:368676				

L20 ANSWER 36 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB A method for easy removal of the copper catalyst
 complex from atom transfer radical polymerization (ATRP) products
 post-polymerization,
 is based on use of precipitons functionalized with ATRP ligands.
 Precipitons, e.g., alc., isocyanate, and amine
 -functionalized polyarom. ligands, were developed to provide a convenient
 method for isolating solutes from homogeneous reaction media. These
 compds. are attached to a reactant and after a reaction is complete they
 can be isomerized to cause precipitation of the attached product. The
 precipiton
 ligands were used in ATRP of Me methacrylate to afford polymers with
 polydispersity of 1.2 - 1.5 and with good control of mol. weight After
 polymerization is complete, the solution was exposed to UV light to
 isomerize the
 precipiton and precipitate the catalyst complex, thus effecting removal
 and subsequent recovery of catalyst. There was no detectable
 copper in the polymer solution, as determined by UV spectroscopy.

ACCESSION NUMBER: 2002:362740 CAPLUS
 DOCUMENT NUMBER: 137:94070
 TITLE: Use of Precipitons for Copper Removal in
 Atom Transfer Radical Polymerization
 Honigfort, Mical E.; Brittain, William J.; Bosanac,
 Todd; Wilcox, Craig S.
 CORPORATE SOURCE: Department of Polymer Science, University of Akron,
 Akron, OH, 44325, USA
 SOURCE: Macromolecules (2002), 35 (13), 4849-4851
 CODEN: MAMOEK; ISSN: 0024-9297
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 37 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The hydrophobic gypsum compns. contain gypsum, the granulated hydrophobic
 additive, and a pH effecting additive in an amount sufficient to maintain pH
 of the composition between 8 and 12.5 in the presence of water. The
 granules
 contain an organopolysiloxane having Si-bonded hydrogen, a water soluble or
 water dispersible binder, and a carrier, preferably gypsum or a stearate
 salt in an amount sufficient to hydrophobize the gypsum. The
 organopolysiloxane is a trialkylsiloxy terminated
 methylhydrogenopolysiloxane with a viscosity of 10-5000 mm²/s at
 250°, and the water dispersible binder is a mixture of an ethoxylated
 fatty alc. and C16-20 fatty acids. The carrier is selected from
 gypsum, calcium sulfate formed in flue gas desulfurization, magnesium
 sulfate or barium sulfate, starch, native starch, Me cellulose,
 CM-cellulose, sand, silica, aluminosilicates, clay materials, calcium
 carbonates, polystyrene beads, polyacrylate beads, ammonium stearate,
 sodium stearate, lithium stearate, potassium stearate, magnesium stearate,
 calcium stearate, barium stearate, zinc stearate, aluminum
 tri-stearate, aluminum distearate, aluminum monostearate and
 copper stearate. The pH effecting additive is preferably lime but
 may be any appropriate additive. The resulting compns. are suitable for
 manufacture of gypsum boards, plasters, and moldings.

ACCESSION NUMBER: 2002:293574 CAPLUS
 DOCUMENT NUMBER: 136:313956
 TITLE: Granulated hydrophobic organopolysiloxane additive for
 gypsum compositions
 Windridge, James; Butler, Derek; Gubbels, Frederic;
 Wehner, Manfred
 INVENTOR(S): Dow Corning Corporation, USA; Dow Corning SA
 PATENT ASSIGNEE(S): PCT Int. Appl., 30 pp.
 SOURCE: CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002030847	A1	20020418	WO 2001-GB4198	20010920
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2001087904	A5	20020422	AU 2001-87904	20010920
EP 1328487	A1	20030723	EP 2001-967531	20010920
EP 1328487	B1	20040618		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
AT 273938	E	20040915	AT 2001-967531	20010920
US 2004050287	A1	20040318	US 2003-398327	20031008
PRIORITY APPLN. INFO.: GB 2000-24642 A 20001007 WO 2001-GB4198 W 20010920				
REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT				

L20 ANSWER 37 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

120 ANSWER 40 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The coating method, useful for automobile bodies, etc., contains (A) applying powder coatings of blocked polyisocyanates and thermostetting resins on electroconductive substrates, (B) heating in the conditions where the resins melt but crosslinking does not occur, (C) electrodepositing comp. (curing temperature 100-150°, pH 5.7-6.7), which comprise blocked polyisocyanates, cationic resins, and metal curing catalysts (metal content 0.1-0.5 parts based on 100 parts solid of the compns.) for the thermostetting resins, so as to cover uncoated area of the substrates, (D) water-washing, and (E) curing at 160-200°. Thus, applying a polyether powder coating containing blocked polyisocyanates (Powdax P 100) on a steel plate, electrodepositing a composition comprising bisphenol A glycidyl ether-diethylamine copolymer, lead acetate trihydrate, and TDI-trimethylolpropane compound blocked with H₂ Et ketoxime, furfuryl alc., and ethylene glycol monobutyl ether, baking, and finally applying an acrylic melamine resin topcoat (Superlac silver and clear) to give a test piece showing adhesion of the coating layers.

ACCESSION NUMBER: 2001:733950 CAPLUS
 DOCUMENT NUMBER: 135:274273
 TITLE: Method for multilayer anticorrosive coatings by powder coating and electrodeposition
 INVENTOR(S): Fukuno, Junichi; Yamaguchi, Masayuki; Ishiwatari, Masaru; Iwasato, Koichi; Ukita, Tsuneo
 PATENT ASSIGNEE(S): Honda Motor Co., Ltd., Japan; Nippon Paint Co., Ltd.
 SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.
 CODEN: JXOXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001276722	A2	20011009	JP 2000-91150	20000329
PRIORITY APPLN. INFO.:			JP 2000-91150	20000329

AB ANSWER 41 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
A family of crystalline meline, silicates "zeolite SSZ-55" is obtained in its silicate, aluminosilicate, or borosilicate form having the following composition: YO_2/WO_3 with a mole ratio of 20-150, $Y = Si, Ge, W, Al, Ga,$
Fe, B, Ti, In, V, C = 1 or 2, d = 2 if C = 1 or d = 3 or 5 if C = 2; $M_2/n/YO_2$ with mole ratio of 0.01-0.03, H = alkali metal cation or alkaline earth cation and n is the valence of M; and Q/YO_2 with a mole ratio of 0.02-0.05 and Q is phenylcycloalkylmethyl ammonium or N-cyclohexyl-N-(2-methylpropyl)pyrrolidinium cation. The zeolite is mainly in the hydrogen form and free of acidity. The zeolite can be used as a catalyst for processes, such as hydrocracking, dewaxing of hydrocarbons, the production of a C20+ lube oil from C20+ olefins, increasing the octane of a hydrocarbon feedstock by conversion of hydrocarbons into aromas, alkylation and transalkylation of aromatic hydrocarbons, isomerization or oligomerization of olefins, and the conversion of lower alcs. and other oxygenated hydrocarbons into liquid products. For the catalytic dewaxing of linear chain and slightly branched hydrocarbons in the presence of hydrogen at a pressure of 15-3000 psi the catalyst consists of a first layer containing the described zeolite and a group VIII metal (platinum) and second layer of an aluminosilicate zeolite which is more shape selective. For the conversion of paraffins into aromas the catalyst contains also gallium, titanium or their mixture. In combination with a Fischer-Tropsch or methanol synthesis catalyst syngas can be converted into mainly liquid hydrocarbons. The zeolite containing also metal or metal ions, such as cobalt or copper, can be used for the reduction of oxides of nitrogen in a gas stream in the presence of oxygen and could be placed in the exhaust stream of an internal combustion engine.

ACCESSION NUMBER: 2001:676697 CAPLUS
DOCUMENT NUMBER: 135:228874
TITLE: Preparation of crystalline zeolite SSZ-55 using quaternary organic amines as a template and its use as a catalyst for the conversion of hydrocarbons
INVENTOR(S): Elomari, Saleh; Harris, Thomas V.
PATENT ASSIGNEE(S): Chevron U.S.A. Inc., USA
SOURCE: PCT Int. Appl., 60 pp.
CODEN: PIXX02
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 5
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001066464	A2	2001051913	WO 2001-056655	20010228
WO 2001066464	A3	200202124		
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BE, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NZ, NL, PT, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
RV:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, JP, KR, MA, NL, PT, SE, TR, BF, BG, CF, CG, CH, CN, CU, GE, GM, HE, HU, NE, SD, TD, TG			
US 6475463	B1	20021105	US 2000-520640	20000307

L20 ANSWER 43 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB Long chain alkyl di-Me amine is an important organic intermediate. Its main preparation process is the catalytic amination of fatty alc. with di-Me amine under the action of hydrogenation-dehydrogenation catalysts. The main improving target of the amination catalysts is to promote their selectivity. The effect of adding Zn to the system of Cu-Ni supported on CaCO₃ and the mechanism of the action of Zn have been studied in this paper. It is found that the selectivity of Cu-Ni catalyst is improved effectively by the adding of Zn, and an important action of Zn is that it can prohibit the reduction of Ni in the catalyst. The reduction level of Ni is responsible for the selectivity of the catalysts.

ACCESSION NUMBER: 2001:174925 CAPLUS
DOCUMENT NUMBER: 134:354808
TITLE: Study on Cu-Ni-Zn catalyst for catalytic amination of fatty alcohol
AUTHOR(S): Li, Qixiao; Zhang, Gaoyong; Peng, Shaoyi
CORPORATE SOURCE: China Research Institute of Daily Chemical Industry, Taiyuan, 030001, Peop. Rep. China
SOURCE: Cuihua Xuebao (2001), 22(1), 7-10
CODEN: THHPD3; ISSN: 0253-9837
PUBLISHER: Kexue Chubanshe
DOCUMENT TYPE: Journal
LANGUAGE: Chinese

L20 ANSWER 42 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB N,N-dimethyl-N-alk(en)ylamine is prepared by amination of higher alc. with Me₂NH at 100-250° and 1-9.8 MPa in the presence of catalyst comprising Cu, the 4th periodic transition metal element (except Cr), and optionally Group VIII Pt-group element, with feeding Me₂NH and H to the reaction system, removing generated H₂O from the system, and adjusting the Me₂NH content 0.5-50 volume% in the H₂O-removed exhaust gas. Feeding of Me₂NH to the system is terminated when the mol ratio of the higher alc./N-methyl-N-alk(en)ylamine byproduct becomes 1-1.5. H, however, is continued to be supplied to the system for continuing the amination. Thus, Kalcobl 80 (stearyl alc.) was aminated with Me₂NH under H in the presence of Cu Ni oxide immobilized on synthetic zeolite at 100° to give N,N-dimethylstearylamine with 99.7% purity.

ACCESSION NUMBER: 2001:406259 CAPLUS
DOCUMENT NUMBER: 135:5372
TITLE: Preparation of high-purity N,N-dimethyl-N-alk(en)ylamine from higher alcohol and dimethylamine
INVENTOR(S): Taniguchi, Hideki; Abe, Hiroshi
PATENT ASSIGNEE(S): Kao Corp., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001151733	A2	20010605	JP 1999-335750	19991126

PRIORITY APPLN. INFO.: JF 1999-335750 19991126
OTHER SOURCE(S): CASREACT 135:5372

L20 ANSWER 44 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB The method comprise heating melamine or its derivs. with alcs. without substantial H gas in the presence of hydrogenation and dehydrogenation catalysts. Thus, melamine was heated with 1,4-butanediol in the presence of Pd/C and Girdler G 96D (Ni-NiO-SiO₂ catalyst) at 220° for 3 h to give 22.0% 2,4-diamino-6-(4-hydroxybutylamino)-1,3,5-triazine, 3.0% 2-amino-4,6-bis(4-hydroxybutylamino)-1,3,5-triazine, and 0.1% 2,4,6-tris(4-hydroxybutylamino)-1,3,5-triazine.

ACCESSION NUMBER: 2001:58467 CAPLUS
DOCUMENT NUMBER: 134:116321
TITLE: Method for modification of melamine derivatives
INVENTOR(S): Nakamura, Shigeo; Kamisaka, Hiroyuki
PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 12 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001019682	A2	20010123	JP 1999-191279	19990706

PRIORITY APPLN. INFO.: JF 1999-191279 19990706
OTHER SOURCE(S): MARPAT 134:116321

L20 ANSWER 45 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Compns. and methods for assaying analytes, preferably, small mol. analytes are provided. Assay methods employ, in place of antibodies or mols. that bind to target analytes or substrates, modified enzymes, called substrate trapping enzymes. These modified enzymes retain binding affinity or have enhanced binding affinity for a target substrate or analyte, but have attenuated catalytic activity with respect to that substrate or analyte. The modified enzymes are provided. In particular, mutant S-adenosylhomocysteine (SAH) hydrolases, substantially retaining binding affinity or having enhanced binding affinity for homocysteine or S-adenosylhomocysteine but having attenuated catalytic activity, are provided. Conjugates of the modified enzymes and a facilitating agent, such as agents that aid in purification or linkage to a solid support are

also provided.
 ACCESSION NUMBER: 2001:31675 CAPLUS
 DOCUMENT NUMBER: 134:83111
 TITLE: Methods and compositions for assaying analytes
 INVENTOR(S): Yuan, Chong-Sheng
 PATENT ASSIGNEE(S): General Atomics, USA
 SOURCE: PCT Int. Appl., 187 pp.
 CODEN: PIXKD2
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001002600	A2	20010111	WO 2000-0518057	20000630
WO 2001002600	A3	20020110		
WO 2001002600	C2	20020725		
W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AN, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RV: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LJ, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CH, GA, GN, GW, HL, HR, KE, SN, TD, TG				
US 6376210	B1	20020423	US 1999-347878	19990706
CA 2377665	AA	20010111	CA 2000-2377665	20000630
GB 2368641	A1	20020508	GB 2002-425	20000630
GB 2368641	B2	20041006		
PRIORITY APPLN. INFO.:		US 1999-347878	A	19990706
		US 1999-457205	A	19991206
		WO 2000-0518057	W	20000630

L20 ANSWER 47 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Asym. conjugate addition of diethylzinc to cyclohexen-2-one, chalcone, and benzalacetone has been found to occur with 0.5% copper(II) triflate and 1% chiral phosphite. Cyclic phosphites derived from TADDOL gave excellent to moderate enantiomeric excesses. The nature of the exocyclic substituent of the dioxaphospholane ring is important, but the chiral induction is imposed by the TADDOL framework. Syntheses of all the TADDOL ligands are described.

ACCESSION NUMBER: 2001:3418 CAPLUS
 DOCUMENT NUMBER: 134:222271
 TITLE: Synthesis and application of chiral phosphorus ligands derived from TADDOL for the asymmetric conjugate addition of diethyl zinc to enones
 AUTHOR(S): Alexakis, Alexandre; Burton, Jonathan; Vastar, Johann; Benhaim, Cyril; Fournioux, Xavier; Van den Heuvel, Alexandra; Leveque, Jean-Marc; Maze, Frederique; Rosset, Stephane
 CORPORATE SOURCE: Department of Organic Chemistry, University of Geneva, Geneva, 1211/4, Switz.
 SOURCE: European Journal of Organic Chemistry (2000), (24), 4011-4027
 CODEN: EJOCFK; ISSN: 1434-193X
 PUBLISHER: Wiley-VCH Verlag GmbH
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 134:222271
 REFERENCE COUNT: 61
 THERE ARE 61 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 46 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The catalyst was prepared by adding dropwise aqueous metal nitrate salt and Na2CO3 solns. to a mixture of CaCO3 and H2O, filtering, drying, and calcining at 400°. The addition of Zn improved the selectivity and decreased the reducibility of Ni of the catalyst compared with Cu-Ni series catalyst, based on the study of amination of lauryl alc. The selectivity of the catalyst decreased with the increasing the content of reduced Ni in the catalyst.

ACCESSION NUMBER: 2001:17377 CAPLUS
 DOCUMENT NUMBER: 135:62914
 TITLE: Cu-Ni-Zn fatty alcohol amination catalyst
 AUTHOR(S): Li, Qixiao; Zhang, Gaoyong; Peng, Shaoyi
 CORPORATE SOURCE: China Institute of Domestic Chemicals, Taiyuan, 030001, Peop. Rep. China
 SOURCE: Xinsbiji De Cuihuo Kexue Yu Jishu, Quanguo Cuihuo Kexue Jihui Lunwenji, 10th, Zhangjiajie, China, Oct. 15-19, 2000 (2000), 239-240. Editor(s): Zhong, Bing. Shanxi Kexue Jishu Chubanshe: Taiyuan, Peop. Rep. China.
 CODEN: 69ASHU
 DOCUMENT TYPE: Conference
 LANGUAGE: Chinese

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000264893	A2	20000926	JP 1999-227497	19990811
PRIORITY APPLN. INFO.:		JP 1999-8170	A	19990114
OTHER SOURCE(S):		MARPAT 133:252562		

L20 ANSWER 49 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Two new Schiff bases (1a)-(2a) derived from 2,3-diaminopyridine (DAPY) with pyrrole-2-carboxaldehyde (Pyr) and 2-hydroxy-1-naphthaldehyde (NaphH), and new Cu(II), Fe(III), Ni(II), Ru(II) and Zn(II) complexes (1b)-(1e), (2b)-(2f) and (3b)-(3d) derived from these two new Schiff bases, and the bis-condensed Schiff base of 2,3-diaminopyridine and salicylaldehyde (SalH) were synthesized. They were characterized by a combination of elemental analyses, magnetic susceptibility measurements, IR and NMR spectra. The two new Schiff bases and some of the metal complexes show antibacterial activity. The Fe(III) and Ru(II) complexes display catalytic activity in the oxidation of alcs. in the presence of N-methylmorpholine-N-oxide as co-oxidant.

ACCESSION NUMBER: 2000:520951 CAPLUS
 DOCUMENT NUMBER: 133:246487
 TITLE: Synthesis and anti-bacterial/catalytic properties of Schiff bases and Schiff base metal complexes derived from 2,3-diaminopyridine

AUTHOR(S): Jeewoth, T.; Li Kam Wah, H.; Bhowon, Minu G.; Ghooorhoo, D.; Babooram, K.

CORPORATE SOURCE: Department of Chemistry, University of Mauritius, Reduit, Mauritius

SOURCE: Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry (2000), 30(6), 1023-1038
 CODEN: SRIMCH; ISSN: 0094-5714

PUBLISHER: Marcel Dekker, Inc.

DOCUMENT TYPE: Journal

LANGUAGE: English

REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 50 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Amino carboxylic acid salts were prepared by oxidation of amino alcs. in an aqueous alkaline reaction medium in the presence of a reduced copper /chroma or copper/zinc spinel as catalyst. The method was applied to the oxidation of ethanol to synthesize iniodiacetic acid disodium salt.

ACCESSION NUMBER: 2000:513660 CAPLUS
 DOCUMENT NUMBER: 133:89797
 TITLE: Preparation of amino carboxylic acids by oxidation of primary amino alcohols

INVENTOR(S): Siebenhaar, Bernd; Rusek, Milos

PATENT ASSIGNEE(S): Novartis A.-G., Switz.; Novartis-Erfindungen Verwaltungsgesellschaft m.b.H.

SOURCE: PCT Int. Appl., 13 pp.
 CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2000043351	A1	20000727	WO 2000-EP434	20000120
W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CZ, CU, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, HR, NE, SN, TD, TG				
CA 2359072	AA	20000727	CA 2000-2359072	20000120
EP 1144359	A1	20011017	EP 2000-906215	20000120
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
BR 2000008178	A	20011106	BR 2000-8178	20000120
JP 2002535301	T2	20021022	JP 2000-594769	20000120
ZA 2001005955	A	20020306	ZA 2001-5955	20010719
US 2002038050	A1	20020328	US 2001-911162	20010723
PRIORITY APPLN. INFO.: CH 1999-127			A 19990122	
OTHER SOURCE(S): MARPAT 133:89797			WO 2000-EP434	W 20000120
REFERENCE COUNT: 1				
				THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 51 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Aliphatic primary alcs., including aliphatic primary alcs. possessing one or more oxygen, nitrogen and/or phosphorus heteroatoms that may be atoms substituting for carbon atoms in the alkyl group or component atoms of substituents on the alkyl group, were converted into salts of carboxylic acids by contacting an alkaline aqueous solution of the primary alc. with a catalyst comprising cobalt, copper, and at least one of cerium, iron, zinc, and zirconium. Diethanolamine, for example, was converted to sodium iminodiacetate by treatment in an aqueous medium containing sodium hydroxide with a catalyst that was obtained by reducing a mixture of cobalt, copper, and zirconium oxides with hydrogen.

ACCESSION NUMBER: 2000:191052 CAPLUS
 DOCUMENT NUMBER: 132:222867
 TITLE: Process for preparing carboxylic acids

INVENTOR(S): Ringer, James William; Holzahn, David Craig; Hucul, Dennis Alexander

PATENT ASSIGNEE(S): Dow Agrosciences LLC, USA

SOURCE: PCT Int. Appl., 18 pp.
 CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2000015601	A1	20000323	WO 1999-US21025	19990914
W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TH				
RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, HR, NE, SN, TD, TG				
CA 2343962	AA	20000323	CA 1999-2343962	19990914
AU 9961434	A1	20000403	AU 1999-61434	19990914
AU 751606	B2	20020822		
US 6229045	B1	20010508	US 1999-395506	19990914
BR 9913670	A	20010605	BR 1999-13670	19990914
EP 1114023	A1	20010711	EP 1999-948206	19990914
EP 1114023	B1	20040526		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
JP 2002524548	T2	20020806	JP 2000-570142	19990914
RU 2222523	C2	20040127	RU 2001-110174	19990914
AT 267797	E	20040615	AT 1999-948206	19990914
ES 2219064	T3	20041116	ES 1999-948206	19990914
US 2001008948	A1	20010719	US 2001-781588	20010205
ZA 2001001753	A	20020301	ZA 2001-1753	20010301
PRIORITY APPLN. INFO.: US 1998-100131P			P 19980914	
OTHER SOURCE(S): MARPAT 132:222867			US 1999-395506	A3 19990914
REFERENCE COUNT: 2			WO 1999-US21025	W 19990914
				THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 52 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Cyclic alcs. are prepared by catalytic hydration of C₆H₂s-2-tRt (R = H, C1-4 alkyl, Ph, cyclohexyl; s = 5-12; t = 1-4) in the presence of crystalline metasilicates containing 21 metals selected from Al, B, Ga, Ti, Cr, Fe, Zn, P, V, and Cu and distilled with basic compds. Cyclohexene was hydrated in the presence of aluminosilicate (25M-5) at 125° under 6 kg/cm² to give a cyclohexene mixts. containing 11.8 weight% cyclohexanol and 18 ppm aluminosilicate, 100 parts of which was distilled with triethylenetetramine to give 11.0 parts cyclohexanol containing 2 weight ppm and 51 weight ppm triethylenetetramine.

ACCESSION NUMBER: 2000:77102 CAPLUS
 DOCUMENT NUMBER: 132:107721
 TITLE: Preparation and isolation of cyclohexanols

INVENTOR(S): Ban, Masakazu; Ishida, Hiroshi

PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
 CODEN: JHXXAF

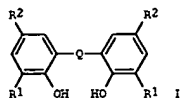
DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000034241	A2	20000202	JP 1998-201784	19980716
PRIORITY APPLN. INFO.: JP 1998-201784				19980716
OTHER SOURCE(S): MARPAT 132:107721				



AB Phenoxyl radicals of tetradentate Cu(II) and Zn(II) complexes of diphenols 1 (Q = S, O, NR3, PR4, or o-NHC6H4NH2; R1, R2 = radical-stabilizing group such as alkyl with a phenolate ring-bonded tertiary C; R3, R4 = H or Cl-6 alkyl) are useful as catalysts for oxidation of primary and secondary alcs. and amines. A typical phenoxyl radical catalyst was manufactured by refluxing THF containing 0.1 g CuCl, 0.44 g 1 (Q = S, R1 = R2 = tert-Bu), and 0.5 mL Et3N 30 min under Ar, cooling to 20°, and treating the solution 1 h with dry O.

ACCESSION NUMBER: 1999:819056 CAPLUS
DOCUMENT NUMBER: 132:65734
TITLE: Cu(II)- and Zn(II)-phenoxide complexes, and radical complexes derived therefrom, their preparation and use

INVENTOR(S): Hess, Martina; Chaudhuri, Phalguni; Wiegardt, Karl
PATENT ASSIGNEE(S): Degussa-Huls A.G., Germany; Degussa A.G.
SOURCE: Eur. Pat. Appl., 14 pp.
CODEN: EPXKDW

DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 967215	A2	19991229	EP 1999-112025	19990622
EP 967215	A3	200010530		
EP 967215	B1	20030402		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
DE 19828492	A1	19991230	DE 1998-19828492	19980626
DE 19925142	A1	20001207	DE 1999-19925142	19990602
US 6153779	A	20001128	US 1999-340410	19990628
PRIORITY APPLN. INFO.: DE 1998-19828492 A 19980626 DE 1999-19925142 A 19990602				
OTHER SOURCE(S): MARPAT 132:65734				

AB The tetradentate ligand N,N'-bis(3,5-di-tert-butyl-2-hydroxyphenyl)-1,2-phenylenediamine, H4L1, has been prepared, and its square planar complexes [CuII(L3)] and [ZnII(L3)] have been synthesized from the reaction of H4L1 with [CuI(NCCH3)4] (ClO4) or Zn (BF4)2·2H2O in methanol in the presence of air. The dianion (L3)2- represents the two-electron oxidized form of (L1)4-, namely N,N'-bis(3,5-di-tert-butyl-2-hydroxyphenyl)-1,2-diiminoquinone. Complexes [CuII(L3)]·CH3CN and [Zn(L3)]·CH3CN have been characterized by X-ray crystallog., ESR spectroscopy, and magnetochem.; [CuII(L3)] has an S = 1/2 ground state, and [Zn(L3)] is diamagnetic. Cyclic voltammetry established that both complexes undergo two successive reversible one-electron oxidns. and two successive reversible one-electron redns. Thus, the coordinated ligand exists in five oxidation levels. The species [MII(L4)]PF6 (M = CuII, ZnII) and [MII(L5)](ClO4)2 (M = CuII, ZnII) have been isolated and characterized by UV/vis, ESR and IR NMR spectroscopy and by magnetic susceptibility measurements, where (L4)- represents the monoanion N-(3,5-di-tert-butyl-2-hydroxyphenyl)-N'-(3,5-di-tert-butyl-2-phenoxy)-1,2-diiminoquinone and (L5) is the neutral ligand N,N'-bis(3,5-di-tert-butyl-2-phenoxy)-1,2-diiminoquinone. Similarly, two complexes of the type [MII(L1H2)] (M = CuII, ZnII) have been isolated from the reaction of L1H4 with CuI(ClO4)2·6H2O or Zn(ClO4)2·6H2O under anaerobic conditions in the presence of NET3. Complexes [CuII(L4)]PF6 and [Zn(L4)]PF6 selectively oxidize primary alcs. (including methanol and ethanol) in a stoichiometric fashion under anaerobic conditions, yielding the corresponding aldehydes and [MII(L2H2)]+ (M = CuII, ZnII), where (L2)3- is the trianion of N,N'-bis(3,5-di-tert-butyl-2-hydroxyphenyl)-1,2-diiminoquinone. Since the latter reduced forms react rapidly with dioxygen with formation of [MII(L4)]+ (M = Cu, Zn) and 1 equiv of H2O2, these oxidized species are catalysts for the air oxidation of primary alcs., including ethanol and methanol, with concomitant formation of H2O2 and aldehydes. The kinetics of the stoichiometric reactions and of the catalyzes (initial rate method) have been measured. Large kinetic isotope effects show that H-abstraction from the α-carbon atom of a coordinated alkoxy ligand is the rate-determining step in all cases.

ACCESSION NUMBER: 1999:629681 CAPLUS
DOCUMENT NUMBER: 132:49684
TITLE: Aerobic Oxidation of Primary Alcohols (Including Methanol) by Copper(II)- and Zinc(II)-Phenoxyl Radical Catalysts
AUTHOR(S): Chaudhuri, Phalguni; Hess, Martina; Mueller, Jochen; Hildebrand, Knut; Bill, Eckhard; Weyhermüller, Thomas; Wiegardt, Karl
CORPORATE SOURCE: Max-Planck-Institut fuer Strahlenchemie, Muelheim a.d. Ruhr, D-45470, Germany
SOURCE: Journal of the American Chemical Society (1999), 121(41), 9599-9610
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

AB Double allylation of carboxylic esters with allyl bromide was performed successfully by the action of Al metal and a catalytic amount of Pb(II) bromide in THF. E.g., PhCH2CO2Me reacted with allyl bromide, Al and PbBr2 catalyst in THF at room temperature to give 98% yield of PhCH2CH(OH)(CH2CH=CH2)2. The proper choice of solvent is essential for the reaction; thus, among the examined solvents, ethers, e.g., THF, 1,2-dimethoxyethane (DME), and Et2O, could be successfully used for the double allylation but with DMF, aqueous MeOH, and aqueous THF, no appreciable reaction occurred. Allylation of benzaldehyde di-Me acetal and N-benzylimine in THF under the same conditions took place smoothly to afford the corresponding allylation products. In a similar manner, allylation of benzonitrile was also performed to afford the doubly allylated benzyamine.

ACCESSION NUMBER: 1999:796180 CAPLUS
DOCUMENT NUMBER: 132:165971
TITLE: Barbier-type allylation of carbonyl derivatives by use of aluminum as an electron pool. Double allylation of carboxylic esters
AUTHOR(S): Tanaka, H.; Nakahata, S.; Watanabe, H.; Zhao, J.; Kuroboshi, M.; Torii, S.
CORPORATE SOURCE: Faculty of Engineering, Department of Applied Chemistry, Okayama University, Okayama, Japan
SOURCE: Inorganica Chimica Acta (1999), 296(1), 204-207
CODEN: ICHAA3; ISSN: 0020-1693
PUBLISHER: Elsevier Science S.A.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 132:165971
REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

AB Substituents are introduced to melamine or N-substituted melamine derivs. by reacting melamine or a melamine derivative with an alc. while heating in the presence of hydrogen and a catalyst mixture including a hydrogenation catalyst and a dehydrogenation catalyst. Comps. obtained by introducing substituents into amino groups of melamine derivs. according to the procedure of this invention are widely usable as fine chemical intermediates in a number of fields including agricultural chems., drugs, dyes, and paints, as well as various resin materials and flame-retardant materials.

ACCESSION NUMBER: 1999:464284 CAPLUS
DOCUMENT NUMBER: 131:102974
TITLE: Modification of melamine derivatives by reaction with alcohols in the presence of hydrogenation and dehydrogenation catalysts
INVENTOR(S): Tanaka, Norio; Kousaka, Hiroyuki; Nakajima, Yasuyuki; Masahashi, Kouichi; Kitabayashi, Takashi
PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan
SOURCE: PCT Int. Appl., 54 pp.
CODEN: PIXKX2
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9936411	A1	19990722	WO 1999-JP123	19990118
W: CA, JP, NO, US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
CA 2319219	AA	19990722	CA 1999-2319219	19990118
EP 1057821	A1	20001206	EP 1999-900332	19990118
EP 1057821	B1	20040526		
R: AT, BE, CH, DE, FR, GB, IT, LI, NL, SE				
AT 267819	E	20040615	AT 1999-900332	19990118
NO 2000003679	A	20000919	NO 2000-3679	20000718
US 6307046	B1	20011023	US 2000-600122	20000731
PRIORITY APPLN. INFO.: JP 1998-7220 A 19980119 JP 1998-8263 A 19980120 WO 1999-JP123 W 19990118				

OTHER SOURCE(S): MARPAT 131:102974
REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 57 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB Polyamines (e.g., ethylenediamine) are prepared with increased selectivity for linear aminated products and with inhibited formation of discoloring byproducts, or byproducts (e.g., piperazine) which may later cause discoloration, by performing, in a first part of the process, the amination of alcs., phenols, diols, or aminoalcs. (e.g., ethanediolamine) with ammonia, primary, or secondary amines in the presence of a hydrogenation/dehydrogenation catalyst (e.g., metallic nickel promoted with Ru, Re, Pd, Pt, or their mixts., on a porous metal oxide support containing alumina) to a conversion degree of between 50-98% (calculated on the total yield of polyamines) at a proportionally time-weighted average temperature, which is at least 15° higher than the proportionally time-weighted average temperature in the remaining part.

ACCESSION NUMBER: 1999:325894 CAPLUS

DOCUMENT NUMBER: 130:325849

TITLE: Process and catalysts for the preparation of linear, nonpolymeric low-color polyamines

INVENTOR(S): Gunther-Hanssen, Johan

PATENT ASSIGNEE(S): Akzo Nobel N.V., Neth.

SOURCE: PCT Int. Appl., 14 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9924389	A1	19990520	WO 1998-SE1772	19981001
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
SE 9704116	A	19990512	SE 1997-4116	19971111
SE 513250	C2	20000807		
CA 2306145	AA	19990520	CA 1998-2306145	19981001
AU 9893721	A1	19990531	AU 1998-93721	19981001
AU 744480	B2	20000228		
EP 1044183	A1	20001018	EP 1998-946779	19981001
EP 1044183	B1	20030205		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, FI				
EE 200000222	A	20010416	EE 2000-200000222	19981001
EE 4430	B1	20050215		
BR 9815223	A	20010821	BR 1998-15223	19981001
JP 2001522824	T2	20011120	JP 2000-520403	19981001
AT 232198	E	20030215	AT 1998-946779	19981001
ES 2191967	T3	20030816	ES 1998-946779	19981001
RU 2215734	C2	20031110	RU 2000-115298	19981001
TW 461901	B	20011101	TW 1998-87116607	19981007
US 5994585	A	19991130	US 1998-190486	19981112
BG 104347	A	20001031	BG 2000-104347	20000417
BG 63598	B1	20020628		
NO 2000002075	A	20000705	NO 2000-2075	20000419
PRIORITY APPLN. INFO.:			SE 1997-4116	A 19971111
			WO 1998-SE1772	V 19981001

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS

L20 ANSWER 58 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB The title powder, useful as catalyst support, is prepared by: (1) colloid preparation: dripping metal salt solution (containing stabilizer) into precipitating agent solution at pH 5.0-9.0 with stirring at 120-480 rpm; (2) phase transfer: dissolving metal surfactant in organic solvent, adding the solution, a synergistic agent, a demulsifying agent, and a defoaming agent to the above colloid, with stirring for 1-20 min at 300-1000 rpm; (3) washing, removing organic solvent and water; (4) drying at 105-160° for 1-8 h; roasting at 300-650° for 2-24 h. The metal surfactant is metal carboxylate such as Cu laurate or Zn laurate; the organic solvent is benzene, xylene, industrial benzene, petroleum ether, or industrial solvent naphtha; the synergistic agent is fatty alc. or fatty amine (C8-8) or dodecanol; the stabilizer is Span 80; and the defoaming agent is C3-8 carboxylic acid or pentanoic acid.

ACCESSION NUMBER: 1999:185958 CAPLUS

DOCUMENT NUMBER: 130:187674

TITLE: Preparation of coated superfine powder with metal surfactant by phase transfer

INVENTOR(S): Chen, Songying; Hu, Zeshan; Gao, Yinben; Peng, Shaoyi

PATENT ASSIGNEE(S): Shanxi Coal Chemical Inst., Chinese Academy of

Sciences, Peop. Rep. China

SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 8 pp.

CODEN: CNDKEV

DOCUMENT TYPE: Patent

LANGUAGE: Chinese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CN 1120978	A	19960424	CN 1994-117468	19941020
CN 1054780	B	20000726		
PRIORITY APPLN. INFO.:			CN 1994-117468	19941020

L20 ANSWER 57 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN (Continued)

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 59 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB A cyclic alc., which is obtained by catalytic hydration of a cyclic olefin phase such as cyclohexene with an aqueous phase using crystalline metallosilicate containing at least one metal selected from Al, B, Ga, Ti, Cr,

Fe, Zn, P, V, and Cu as the catalyst, is separated from the oil phase using a distillation tower, wherein a basic substance

such as amine is added to the oil phase after the catalytic hydration before or during the distillation. In prior art, a very small amount of a metallosilicate is carried over to the oil phase and concentrated in cyclic alcs. and catalyzes dehydration of cyclic alcs. to olefins at the beginning of the distillation or even during storage and results

in continuous increase in the olefin concentration. Addition of a very small amount of

a basic substance (e.g. triethylene tetramine) to the oil phase in the distillation deactivates the metallosilicate catalyst (e.g. ZSM-5 or gallosilicate) and decreases the cyclic olefin concentration in the cyclic alc. without lowering the yield of the cyclic alc. and gives cyclic alc. of high purity.

ACCESSION NUMBER: 1999:58432 CAPLUS

DOCUMENT NUMBER: 130:153407

TITLE: Method for isolating and obtaining cyclic

alcohol by distillation

INVENTOR(S): Ban, Masakazu; Ishida, Hiroshi

PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JIOKAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 11012209	A2	19990119	JP 1998-56581	19980309
PRIORITY APPLN. INFO.:			JP 1997-112052	A 19970430
OTHER SOURCE(S):			MARPAT 130:153407	

L20 ANSWER 60 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The title reaction was examined over Pt-containing catalysts (Pt-Al₂O₃, Pt-In₂O₃-Al₂O₃, Pt-ZnO-Al₂O₃). Catalysts with suppressed acidity were the most selective. A new mechanistic scheme for the reaction was proposed and substantiated by means of tracer and kinetic studies. The scheme includes dehydrogenation of the initial amino alc. and intermediate formation of methyltetrahydropyrazine. The scheme was helpful in designing the catalyst. Copper-containing catalysts showed high selectivity and stability in methylpyrazine synthesis. Some of them, tested in prolonged runs at 350°, gave methylpyrazine with yields up to 83%.

ACCESSION NUMBER: 1998:715079 CAPLUS
 DOCUMENT NUMBER: 130:81485
 TITLE: Synthesis of methylpyrazine from N-(2-hydroxypropyl)-1,2-ethanediamine. Mechanistic study and catalyst selection
 AUTHOR(S): Isagulyants, G. V.; Gitis, K. M.
 CORPORATE SOURCE: N.D.Zelinsky Institute of Organic Chemistry (Russian Academy of Sciences), Moscow, 117913, Russia
 SOURCE: Chemical Industries (Dekker) (1998), 75(Catalysis of Organic Reactions), 443-454
 CODEN: CHEIDI; ISSN: 0737-8025
 PUBLISHER: Marcel Dekker, Inc.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 130:81485
 REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 61 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB (N-substituted) amines are prepared by treatment of NH₃, primary amines, or secondary amines with (i) alc. or (ii) aldehydes and H in the presence of catalysts prepared by reduction of malachite- and Al(OH)₃-free precursors containing Cu, Al, and Cr, Mn, Fe, Co, Ni, and/or Zn. Aqueous solution containing Cu(NO₃)₂, Ni(NO₃)₂, and Al(NO₃)₃ was treated with aqueous Na₂CO₃ to give hydrotalcite-like substance, which was reduced in lauryl alc. NPM₂ and H were passed through the catalyst-containing reactor at 200° over 10 h to give colorless products containing 91.8% lauryldimethylamine.

ACCESSION NUMBER: 1998:693414 CAPLUS
 DOCUMENT NUMBER: 129:275633
 TITLE: Metal catalysts and preparation of the catalysts and (N-substituted) amines
 INVENTOR(S): Muraiishi, Teruo; Kato, Kozo
 PATENT ASSIGNEE(S): Mitsui Chemicals Inc., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
 CODEN: JKKKAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10287628	A2	19981027	JP 1997-89526	19970408

PRIORITY APPLN. INFO.: JP 1997-89526 19970408

L20 ANSWER 62 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Porous articles are immersed in and/or coated with organic compds. having CH₂:CH, CH:CH, CH₂:CH₂, CH₂:N, CH:N, NH:CH, NH:N and/or N:N (A), B-containing compds. (B), and polymerization catalysts (C) and polymerized Compns. for the porous articles comprise A, B, and C. Thus, 1% saponified polymer prepared from acetal protected 4-allyl-1,2-dihydroxybenzene and vinyl acetate, 0.6% boric acid, 2% ligninsulfonic acid, 30 ppm polyphenol oxidase was blended to give a solution, in which a chip of wood was immersed. After the solution in the chip was oxidized, polymerized, and mixed with H₂O, 8% boric acid flowed out from the chip.

ACCESSION NUMBER: 1998:650992 CAPLUS
 DOCUMENT NUMBER: 129:332238
 TITLE: Treatment of porous articles and microbiocidal and insect-repellent compositions containing boron compounds for them
 INVENTOR(S): Aoki, Hiroshi; Tanaka, Kazumi; Echigo, Takashi
 PATENT ASSIGNEE(S): Showa Denko K. K., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 27 pp.
 CODEN: JKKKAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10265508	A2	19981006	JP 1997-75267	19970327

PRIORITY APPLN. INFO.: JP 1997-75267 19970327

L20 ANSWER 63 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB N-alkylated amines are prepared by reaction of alc. with alkylamines or dialkylamines in the presence of H₂ and Cu and Mg silicate catalysts containing 0-2 weight% BaO, Cr₂O₃, and/or ZnO. Thus, MeNH₂ and HOCH₂CH₂CH₂OH were hydrogenated 24 h at 240° over a CuO/Mg silicate/BaO/Cr₂O₃/ZnO catalyst to give N-methylmorpholine with 77% selectivity.

ACCESSION NUMBER: 1998:608419 CAPLUS
 DOCUMENT NUMBER: 129:202948
 TITLE: Catalytic N-alkylation of amines with alcohols
 INVENTOR(S): Simon, Joachim; Becker, Rainer; Lebkucher, Rolf
 PATENT ASSIGNEE(S): BASF A.-G., Germany
 SOURCE: Eur. Pat. Appl., 6 pp.
 CODEN: EFXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 863140	A1	19980909	EP 1998-103813	19980304
EP 863140	B1	20040602		

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, MC, PT, IE, SI, LT, LV, FI, RO

DE 19709488	A1	19980910	DE 1997-19709488	19970307
JP 10279569	A2	19981020	JP 1998-51964	19980304
US 5917039	A	19990629	US 1998-34276	19980304
CA 2228613	AA	19980907	CA 1998-2228613	19980305
CN 1194973	A	19981007	CN 1998-108038	19980306
CN 1083837	B	20020501		

PRIORITY APPLN. INFO.: DE 1997-19709488 A 19970307
 OTHER SOURCE(S): CASREACT 129:202948
 REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 64 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB To impart antibacterial activity to cellulose-containing fabrics, easy care finishing treatments were carried out using N-methylol crosslinking agents and polyvinyl alc. (PVOH) softening agents in presence of certain polyvalent metal-salt catalysts under a variety of conditions. From the enhancement in the N content, resiliency as well as antibacterial activity imparted to the finished samples were determined by

the nature of the metal salt-catalyst and followed the decreasing order: $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} > \text{ZnSO}_4 \cdot 7\text{H}_2\text{O} > \text{NiSO}_4 > 3 \text{ CdSO}_4 \cdot 8\text{H}_2\text{O} > \text{None}$. Incorporation of PVOH (up to 10 g/l) in the finishing formulation brings about a slight decrease in both the N content and dry wrinkle recovery along with a significant improvement in both the wet wrinkle recovery and the rot-proofing properties of treated samples, reflecting its ability to interact, bind and/or entrap the polyvalent-metal cations and to form a tough elastic film affixed to fiber surface. Inclusion of softening agent (20 g/l) brings about a significant enhancement in softness degree and resiliency as well as resistance to microbiol. deterioration, regardless of the type of softener used. The increase in bound N, retained strength, as well as rot-proofing properties, as a function of the type of crosslinker used, follows the descending order: $\text{MMF} > \text{DMDEHU}$. The rot-proofing properties of the treated samples as a function of type of acid component of the metal-salt catalyst used, follows the descending order: $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} > \text{CuCl}_2 > \text{Cu}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$. Presence of a polyester component in the fabric structure enhances the resiliency and tensile strength as well as rot-proofing properties.

ACCESSION NUMBER: 1998:602200 CAPLUS
 DOCUMENT NUMBER: 129:344419
 TITLE: New approach for imparting antibacterial activity to cellulose-containing fabrics
 AUTHOR(S): Ibrahim, N. A.; Abo-Shosha, M. H.; Gaffar, M. A.
 CORPORATE SOURCE: Textile Research Division, National Research Centre, Cairo, Egypt
 SOURCE: Colourage (1998), 45(7), 13-14, 16-19, 30
 CODEN: COLOBG; ISSN: 0010-1826
 PUBLISHER: Colour Publications Pvt. Ltd.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 REFERENCE COUNT: 40 THERE ARE 40 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 65 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Polyvalent alcs. (e.g., neopentyl glycol) are prepared by the aldol condensation reaction of an aldehyde having an α -hydrogen RCHO ($\text{R} = \text{C}_1\text{-12 alkyl, cycloalkyl, aryl, C}_5\text{14 aralkyl}$) (e.g., isobutyraldehyde) with itself or with a second aldehyde R_2CHO ($\text{R}_2 = \text{H, C}_1\text{-12 alkyl, cycloalkyl, aryl and aralkyl}$) (e.g., formalin) in the presence of a weak-base anion exchanger (e.g., Amberlyst IRA 67) followed by hydrogenation of the intermediate hydroxyaldehyde (e.g., hydroxyisovaldehyde) in the presence of a solvent and a transition-metal hydrogenation catalyst.

ACCESSION NUMBER: 1998:485027 CAPLUS
 DOCUMENT NUMBER: 129:95826
 TITLE: Process and catalysts for the preparation of polyhydric alcohols by the aldol condensation of aldehydes followed by hydrogenation
 INVENTOR(S): Paatero, Erkki; Nummi, Esa; Lindfors, Lars Peter; Nousiainen, Hannu; Hietala, Jukka; Lahtinen, Leila; Haakana, Rami
 PATENT ASSIGNEE(S): Neste Oy, Finland
 SOURCE: PCT Int. Appl., 34 pp.
 CODEN: PIXKD2
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9829374	A1	19980709	WO 1997-F1835	19971230
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, BG, GE, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
FI 9605268	A	19980701	FI 1996-5268	19961230
FI 102474	B1	19981215		
ZA 9711667	A	19980701	ZA 1997-11667	19971229
AU 9853240	A1	19980731	AU 1998-53240	19971230
EP 948476	A1	19991013	EP 1997-950213	19971230
EP 948476	B1	20030611		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE, MC, PT, IE				
CN 1242760	A	20000126	CN 1997-181173	19971230
CN 1092175	B	20021009		
BR 9714443	A	20000321	BR 1997-14443	19971230
JR 2001507356	T2	20010605	JP 1998-529659	19971230
AT 242757	B	20030615	AT 1997-950213	19971230
ES 2201334	T3	20040316	ES 1997-950213	19971230
TW 446695	B	20010721	TW 1998-87102323	19980219
US 6255541	B1	20010703	US 1999-319572	19990609
PRIORITY APPLN. INFO.:			FI 1996-5268	A 19961230
OTHER SOURCE(S):		MARPAT 129:95826	WO 1997-F1835	W 19971230
REFERENCE COUNT:		4		
				THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 66 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Raney nickel catalyst was modified with 0.5 wt% V or Mg to increase the selectivity to secondary amines in the alkylation of ammonia with PROH or i-BuOH. Selectivities of .apprx.70-80% were obtained at 90-95% conversion. The mixed secondary alkylamine, EtBuNH was prepared from EtNH₂ and BuOH on a com. CuO/ZnO/Al₂O₃ catalyst. The highest yield, .apprx.76%, was attained at 190° and EtNH₂/BuOH molar ratio 25.

ACCESSION NUMBER: 1998:375128 CAPLUS
 DOCUMENT NUMBER: 129:96818
 TITLE: Preparation of symmetrical and mixed secondary alkylamines over raney nickel and supported copper catalysts
 AUTHOR(S): Gobolos, S.; Hagedus, M.; Talas, E.; Margitfalvi, J. L.
 CORPORATE SOURCE: Central Research Institute for Chemistry of the Hungarian Academy of Sciences, Budapest, 1525, Hung.
 SOURCE: Studies in Surface Science and Catalysis (1997), 108(Heterogeneous Catalysis and Fine Chemicals IV), 131-138
 CODEN: SSCIDM; ISSN: 0167-2991
 PUBLISHER: Elsevier Science B.V.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 REFERENCE COUNT: 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 67 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Ru-, Ni- and/or Co-containing catalysts used for the title purpose, especially for amination of H₂NCH₂CH₂OH with NH₃ to manufacture H₂NCH₂CH₂NH₂ with improved selectivity, comprise 6-50% (based on total catalyst weight) Co and/or Ni, 0.001-25% Ru, 0-10% Cu and also 0.5% Fe, Rh, Pd, etc., as catalyst promoters, on a metal oxide support. The catalysts are free from corrosive chlorides and having improved stability in continuous operation. A typical catalyst (preparation given) contained Ru 1, Ni 7.9, Co 7.9 and Cu 3.2% on Al₂O₃ support.

ACCESSION NUMBER: 1998:300824 CAPLUS
 DOCUMENT NUMBER: 128:323139
 TITLE: Catalysts for amination of alkylene oxides, alcohols, aldehydes and ketones
 INVENTOR(S): Wulff-Doring, Joachim; Melder, Johann-Peter; Schulz, Gerhard; Volt, Guido; Gutschoven, Frank; Harder, Wolfgang
 PATENT ASSIGNEE(S): Basf A.-G., Germany
 SOURCE: Eur. Pat. Appl., 8 pp.
 CODEN: EPXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 839575	A2	19980506	EP 1997-118717	19971028
EP 839575	A3	19980812		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
DE 19645047	A1	19980507	DE 1996-19645047	19961031
US 595825	A	19990928	US 1997-955121	19971021
JP 10174875	A2	19980630	JP 1997-296886	19971029
CN 1185995	A	19980701	CN 1997-122818	19971031
PRIORITY APPLN. INFO.:			DE 1996-19645047	A 19961031
OTHER SOURCE(S):		MARPAT 128:323139		

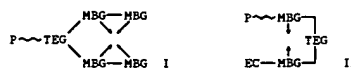
L20 ANSWER 68 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Ru-, Ni- and/or Co-containing catalysts are used for the title
 purpose, especially for amination of H₂NCH₂CH₂OH with NH₃ to manufacture
 H₂NCH₂CH₂NH₂
 with improved selectivity. The catalysts contain decreased
 amts. of Ni or Co and have improved stability in continuous operation. A
 typical catalyst (preparation given) contained Ru 1, Ni 0.79, Co 0.79
 and Cu 1.64 on Al₂O₃ support.

ACCESSION NUMBER: 1998:300823 CAPLUS
 DOCUMENT NUMBER: 128:323138
 TITLE: Catalysts for amination of alkylene oxides,
 alcohols, aldehydes and ketones
 INVENTOR(S): Wulff-Doring, Joachim; Melder, Johann-Peter; Schulz,
 Gerhard; Voit, Guido; Gutschoven, Frank; Harder,
 Wolfgang
 PATENT ASSIGNEE(S): Basf A.-G., Germany
 SOURCE: Eur. Pat. Appl., 9 pp.
 CODEN: EPXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 839574	A2	19980506	EP 1997-118631	19971027
EP 839574	A3	19980812		
EP 839574	B1	20010530		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
DE 19644107	A1	19980507	DE 1996-19644107	19961031
US 5916838	A	19990629	US 1997-955264	19971021
ES 2158423	T3	20010901	ES 1997-118631	19971027
JP 10174874	A2	19980630	JP 1997-296775	19971029
CN 1181284	A	19980513	CN 1997-121251	19971030
CN 1124179	B	20031015		
US 6046359	A	20000404	US 1999-262262	19990304
PRIORITY APPLN. INFO.:			DE 1996-19644107	A 19961031
			US 1997-955264	A3 19971021
OTHER SOURCE(S):	MARPAT 128:323138			

L20 ANSWER 69 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 RV: GH, KE, LS, MW, SD, SE, UG, ZW, AT, BE, CH, DE, DK, ES, FI, FR,
 GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA,
 GN, ML, MR, NE, SN, TD, TG
 AU 9745851 A1 19980414 AU 1997-45851 19970919
 US 6489093 B1 20021203 US 1997-933714 19970919
 PRIORITY APPLN. INFO.: US 1996-26432P P 19960920
 WO 1997-US16740 W 19970919
 REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS
 RECORD: ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 69 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 GI



AB The present invention provides methods and compns., i.e. synthetic
 libraries of binding moieties, for identifying compds. which bind to a
 metal atom or to non-metal ions, e.g., cationic or anionic ions. Thus,
 combinatorial libraries, e.g. I and II (P = TentaGel S amino resin polymer
 support; TEG = turn element group, i.e. di- or trifunctional cyclic amino
 alc. or cyclic amino acid; MBG = metal binding group, i.e. amino
 acid residues; EC = end capping group, i.e. acyl residue) were prepared and
 examined for their ability to coordinate transition metal ions. Thus, a
 12,000 member combinatorial library P-MHCO(CH₂)₅NH-A-B-C-D (III; P-NH₂ =
 TentaGel S amino resin polymer; A (position 1) = L- or D-Asp(OMe₃), L- or
 D-Ser(OMe₃), L- or D-Met, L- or D-Tyr(OMe₃), L- or D-phenylglycine,
 His(CPh₃), Gly; C (position 2) = L-Asp(OMe₃), L-Ser(OMe₃), L-Tyr(OMe₃),
 L-His(CPh₃), L-Met, L-Trp, Gly, L-phenylglycine, 4-piperidinedicarboxylic
 acids; B (turn element) = 1-amino-2-carboxyloxycyclopentane stereoisomers,
 1-amino-2-carboxyloxycyclohexane stereoisomers, 1-amino-2-
 carbonyloxycyclopentane stereoisomers, L-Pro, D-pipecolic acid; D (end cap) =
 RCO, tosyl, pyroglutamic acid, R = Me, OMe₃, 1-naphthyl, CH₂CO₂Me,
 2-pyridyl, 3,4-methylenedioxypheyl, PhNH) was prepared using standard
 solid-phase peptide coupling techniques. Library III was tested for Ni²⁺
 binding affinity by treatment with 2.5 + 10⁻⁴ M Ni(OAc)₂ in MeOH
 followed by solution of dimethylglyoxime in MeOH to form a reddish-pink
 precipitate

trapped in the polymer matrix of about 6 of the 24,000 beads. Tag
 photolysis and anal. allowed the identification of the individual
 nickel-binding library members.

ACCESSION NUMBER: 1998:197471 CAPLUS
 DOCUMENT NUMBER: 128:265374
 TITLE: Combinatorial approach for generating novel
 coordination complexes
 INVENTOR(S): Jacobsen, Eric N.; Francis, Matthew B.; Finney,
 Nathaniel S.
 PATENT ASSIGNEE(S): President and Fellows of Harvard College, USA;
 Jacobsen, Eric N.; Francis, Matthew B.; Finney,
 Nathaniel S.
 SOURCE: PCT Int. Appl., 89 pp.
 CODEN: PIXKDX
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9812156	A1	19980326	WO 1997-US16740	19970919
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				

L20 ANSWER 70 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB A metal paste that can form a coating consists of an organic or inorg. metal
 compound which is in a solid form at normal temperature and an amino
 compound as a
 catalyst. The preferred metal compound is a nitrate, a cyanide, a
 carbonyl compound, or an organic salt compound. The metal is Pd, Pt, Rh,
 Au, Ag,
 Co, Pb, Cu, In, Sn, Sb, Ru, Cd, Tl, Bi, Cr, Mn, Fe, Ni,
 Zn, or Mo. The amino compound is an aromatic or aliphatic
 monoamine or diamine. The paste optionally contains
 aliphatic or aromatic dicarboxylic acids or their esters, mono- or
 polyhydric
 alc., ketones, or ethers. The paste is suitable for firing at a
 low temperature

ACCESSION NUMBER: 1998:178257 CAPLUS
 DOCUMENT NUMBER: 128:273868
 TITLE: Metal paste with wide range of application
 INVENTOR(S): Takamatsu, Hideki
 PATENT ASSIGNEE(S): Nippon Terpen Kagaku K. K., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 12 pp.
 CODEN: JXKXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10072673	A2	19980317	JP 1997-113076	19970430
TW 380146	B	20000121	TW 1997-86105632	19970429
PRIORITY APPLN. INFO.: JP 1996-109563 A 19960430				

L20 ANSWER 71 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Raney nickel, catalyst was modified with 0.5 wt % V or Mg to increase the selectivity to secondary amines in the alkylation of ammonia with n-propanol or i-butanol. Due to the modification selectivities around 70-80% were obtained at 90-95 % conversions. A mixed secondary alkylamine, N-ethyl-N-n-butylamine was prepared from ethylamine and n-butanol on a com. Cu-ZnO-Al₂O₃ (LK-801) catalyst. The highest yield of EtNH-n-Bu around 76% was obtained at 190 °C and EtNH₂/n-BuOH molar ratio 5 or above. In the alkylation of ethylenediamine (EDA) with methanol over LK-801 catalyst monomethyl-EDA was formed with 86% selectivity at T = 185°C, n=3.1, (MeOH/EDA molar ratio) and 51% conversion. Sym. dimethyl-EDA was obtained with 45 % yield at T = 200°C, n = 7.4 and 99% conversion. Tri-Me EDA was prepared with 80% selectivity at T = 215°C, n = 7.4 and complete EDA conversion. In the alkylation of n-butylamine with methanol over LK-801 catalyst correlation has been found between the activity and the ionic copper content of the catalyst. Based on this correlation it has been suggested that in this reaction ionic copper species are involved in the rate determining step, i.e. in the dehydrogenation of methanol into an aldehyde intermediate.

ACCESSION NUMBER: 1998:160156 CAPLUS
 DOCUMENT NUMBER: 128:229959
 TITLE: Preparation of aliphatic secondary mono- and diamines over nickel- and copper-containing catalysts

AUTHOR(S): Gobolos, S.; Margitfalvi, J. L.
 CORPORATE SOURCE: Central Research Institute for Chemistry of the Hungarian Academy of Sciences, Budapest, 1025, Hung.
 SOURCE: Progress in Catalysis (1997), 6(2), 123-134
 CODEN: POCTEU; ISSN: 1220-8698
 Zecasin
 PUBLISHER: Journal
 DOCUMENT TYPE: English
 LANGUAGE: English
 REFERENCE COUNT: 6
 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 72 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The polymeric meso-tetra-(4,4'-biphenylene-bisulfo)-phenylporphyrin and its complexes (PMTBPBSOPP, M = Co, Mn, Cu, and Zn) have been prepared in the presence of phase-transfer catalyst and characterized with UV-visible spectra, IR spectra, XPS, and SEM. Its catalytic activity in oxidation of cumene, ethylbenzene, and cyclohexene by mol. oxygen have been studied. Cumene afforded efficiently cumenol, acetophenone, together with a small amount of 4-Me acetophenone. Ethylbenzene afforded efficiently α -methylbenzyl alc. (4) and acetophenone. Cyclohexene catalyzed by PMTBPBSOPP in the presence of mol. oxygen afforded 1,2-cyclohexanediol and 1,2-cyclohexanedione.

ACCESSION NUMBER: 1998:130980 CAPLUS
 DOCUMENT NUMBER: 128:205371
 TITLE: Sheet polymer and its complexes. II. Preparation and catalytic activity of polymeric tetrakisphenylporphyrin films crosslinked by 4,4'-biphenylene-bisulfonate

AUTHOR(S): Wang, Rong-Min; Li, Shu-Ben; Wang, Yun-Pu; He, Yu-Feng; Lei, Zi-Qiang
 CORPORATE SOURCE: Department of Chemistry, Northwest Normal University, Lanzhou, 730070, Peop. Rep. China
 SOURCE: Journal of Applied Polymer Science (1998), 67(12), 2027-2034
 CODEN: JAPNAB; ISSN: 0021-8995
 PUBLISHER: John Wiley & Sons, Inc.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 REFERENCE COUNT: 15
 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 73 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Acacia and Cryptomeria trees have large plantation areas in the East Asia. We studied the synthesis of biodegradable polyurethane (PU) foams from their bark as its valuable utilization. (+)-Catechin, a model of bark tannin, easily reacted with Ph isocyanate to produce two major urethane derivs. Surprisingly, both of them were formed by the reaction of phenolic, not alc., hydroxyl groups at the 3'- and/or 4'-positions in the B-ring of catechin. Steric hindrance may have affected the reactivity of hydroxyl groups in catechin during the urethane forming reactions. Besides diisocyanates and synthetic polyols, bark of Acacia and Cryptomeria was successfully incorporated into PU foams using triethylenediamine or dibutyltin laurate as catalysts and water as a foaming reagent. The foams had d. of 0.02-0.4 g/cm³ and specific strength of 10-3000 kPa·g·l·cm³. They were biodegradable with some wood-rotting fungi and soil microorganisms, and adsorbed some heavy metal ions, probably due to tannin and other bark components present as ingredients in the foams.

ACCESSION NUMBER: 1997:793273 CAPLUS
 DOCUMENT NUMBER: 128:36109
 TITLE: Biodegradable polyurethane from Acacia and Cryptomeria bark

AUTHOR(S): Sakai, Kokki; Ge, Jin-Jie; Nakashima, Yoji
 CORPORATE SOURCE: Department of Forest Products, Faculty of Agriculture, Kyushu University, Fukuoka, 812-81, Japan
 SOURCE: International Symposium on Wood and Pulp Chemistry, 8th, Helsinki, June 6-9, 1995 (1995), Volume 1, 661-668. Gummerus Kirjapaino Oy: Jyväskylä, Finland.
 CODEN: GSKDAY
 DOCUMENT TYPE: Conference
 LANGUAGE: English
 REFERENCE COUNT: 7
 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 74 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The mechanism for amination of n-octanol (I) with dimethylamine to manufacture dimethyloctylamine (II) was described. The amination was carried out in a fixed bed in the presence of Cu/Cr or Cu/Zn/Cr catalyst. The conversion of I was >99%, and the selectivity of II was >94%. Effects of the preparation of catalysts on their performance and the amination conditions were studied.

ACCESSION NUMBER: 1997:735579 CAPLUS
 DOCUMENT NUMBER: 127:347929
 TITLE: Theory and practice of amination of fatty alcohol with dimethylamine

AUTHOR(S): Qian, Xia; Zhang, Gaocong
 CORPORATE SOURCE: Inst. Daily Chem. Industry, Taiyuan, 030001, Peop. Rep. China
 SOURCE: Riyoung Huaxue Gongye (1997), (2), 4-8
 CODEN: RHGOES; ISSN: 1001-1803
 PUBLISHER: Qinggongyebu Kexue Jishu Qingbao Yanjiuso
 DOCUMENT TYPE: Journal
 LANGUAGE: Chinese

L20 ANSWER 75 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A polymeric alc., such as poly(oxy)alkylene alc. or polyalkyl alc., is reacted at a temperature of 230-380° in the presence of a hydrogenation/dehydrogenation catalyst to produce a polymeric carbonyl intermediate, and the polymeric carbonyl intermediate is reacted with a polyamine, such as ethylenediamine, at a temperature of 150-180° in the presence of hydrogen and a hydrogenation catalyst to produce a polyamine adduct.

ACCESSION NUMBER: 1997:528661 CAPLUS

DOCUMENT NUMBER: 127:123936

TITLE: Reductive amination process for manufacturing a fuel additive from polyoxybutylene alcohol with ethylenediamine

INVENTOR(S): Gray, James A.

PATENT ASSIGNEE(S): Chevron Chemical Company, USA

SOURCE: Eur. Pat. Appl., 13 pp.

CODEN: KPIXKW

DOCUMENT TYPE: Patent

LANGUAGE: English

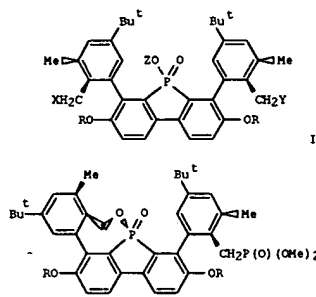
FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 781793	A1	19970702	EP 1996-309310	19961219
EP 781793	B1	20010509		
R: DE, FR, GB, NL				
CA 2191919	AA	19970620	CA 1996-2191919	19961203
JP 09221545	A2	19970826	JP 1996-338627	19961218
PRIORITY APPLN. INFO.:			US 1995-574485	A 19951219

L20 ANSWER 76 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

GI



AB Several improvements, including a generally applicable method for reduction of

aromatic nitro compds. to amines, were made to the preparation from 2,2',4,4'-tetranitrobiphenyl of the meso atropisomer I (R = Ac; X = Y = P(O)(OMe)2; Z = Me) of a bis-phosphonomethylated 4,6-diaryldibenzophosphole 5-oxide, previously obtained in impure form. A concomitant product II (R = Ac) containing one phosphonomethyl group was formed by a novel intramol. displacement. Both products were converted by a specially developed method into crystalline phosphinic-polyphosphonic acids I

(R = CH2PO3H2; X = PO3H2, H; Y = PO3H2; Z = H), containing resp. four and three phosphonomethyl groups, which formed stable monodisperse solns. in H2O at pH 2-4. These solns. catalyzed the hydration of 2-methylpropene to tert-Bu alc. somewhat more efficiently than a toluene-4-sulfonic acid solution of equivalent acidity.

ACCESSION NUMBER: 1997:56529 CAPLUS

DOCUMENT NUMBER: 126:157545

TITLE: Synthesis of substituted dibenzophospholes. Part 9.

Preparation of two water-soluble phosphinic-polyphosphonic acids

Cornforth, John

Sch. Chem. Phys. Environ. Sci., Univ. Sussex,

Brighton, BN1 9QJ, UK

SOURCE: Journal of the Chemical Society, Perkin Transactions

1: Organic and Bio-Organic Chemistry (1996), (24),

2889-2893

CODEN: JCPRB4; ISSN: 0300-922X

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

L20 ANSWER 76 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

OTHER SOURCE(S): CASREACT 126:157545

REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L20 ANSWER 77 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB The amination catalysts comprise 0.001-25% Ru and 0-5% promoters chosen from Fe, Rh, Pd, Pt, Ir, Os, Cu, Ag, Au, Cr, Mo, W, Re, Zn, Cd, Pb, Mn, Sn, Li, Na, K, Rb, Cs, P, As, Sb, Bi, Te, Tl, or their mixts. on porous oxides. The catalysts have good mech. stability. Thus, a 28 Ru-containing alumina catalyst was prepared and used to convert ethanolamine to ethylenediamine with 74.60% selectivity and 52.10% conversion and was still operative after 250 h and feed rate 0.45 kg/L. A prior-art catalyst containing Ni and Ru operating at feed rate 0.33 kg/L had selectivity 61.80% and conversion 33.00% and failed after 48 h.

ACCESSION NUMBER: 1996:628393 CAPLUS

DOCUMENT NUMBER: 125:250945

TITLE: Preparation of amines from alcohols, ketones and aldehydes and amination catalysts therefor

INVENTOR(S): Melder, Johann-Peter; Wulff-Doering, Joachim;

Gutschoven, Frank; Witzel, Tom; Harder, Wolfgang

PATENT ASSIGNEE(S): BASF A.-G., Germany

SOURCE: Ger. Offen., 6 pp.

CODEN: GWXXEX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19507007	A1	19960829	DE 1995-19507007	19950228
EP 729785	A1	19960904	EP 1996-102407	19960217
R: BE, DE, FR, GB, IT, NL, SE				
JP 08243392	A2	19960924	JP 1996-39759	19960227
CN 1138499	A	19961225	CN 1996-103465	19960228
PRIORITY APPLN. INFO.:			DE 1995-19507007	A 19950228
OTHER SOURCE(S):			MARPAT 125:250945	

L20 ANSWER 78 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The reaction of primary alkyl bromides or chlorides with diethylzinc in the presence of Ni(acac)₂ (5 mol %) furnishes the corresponding alkylzinc halides (X = Br, Cl) via a halogen-zinc exchange reaction. The treatment of terminal alkenes with diethylzinc (neat, 25-60 °C) in the presence of Ni(acac)₂ as a catalyst (1-10 mol %) and 1,5-cyclooctadiene (COD) affords the corresponding dialkylzincs via a hydrozincation reaction. Whereas the conversion for simple alkenes bearing a remote functionality reaches 40-63%, the hydrozincation of allylic, homoallylic alcs. and allylic amines proceeds very efficiently (85-95% conversion). All the zinc organometallics obtained react with various electrophiles (allylic halides, enones, acid chlorides, alkynyl halides, Et propiolate) after transmetalation with CuCN·2LiCl. In the presence of the chiral catalyst [(R,R)-1,2-bis(trifluoromethanesulfonamido)cyclohexane], the dialkylzincs prepared add to aldehydes with high enantioselectivity. Thus, Ni(acac)₂-catalyzed reaction of octyl bromide with Et₂Zn in Et₂O/hexane followed by sequential treatment with CuCN·2LiCl in THF and Et (2-bromomethyl)acrylate gave 79% Et 2-nonylacrylate.

ACCESSION NUMBER: 1996:590408 CAPLUS
 DOCUMENT NUMBER: 125:246868
 TITLE: Nickel-Catalyzed Preparations of Functionalized Organozincs
 AUTHOR(S): Vettel, Stephan; Vaupel, Andrea; Knochel, Paul
 CORPORATE SOURCE: Fachbereich Chemie der Philipps-Universität Marburg, Marburg, D-35032, Germany
 SOURCE: Journal of Organic Chemistry (1996), 61(21), 7473-7481
 CODEN: JOCEAH; ISSN: 0022-3263
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 125:246868

L20 ANSWER 79 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Six catalysts (CuO/ZnO/Al₂O₃, 100Fe₃O₄/5V₂O₅/0-4.2BaO/1-1.5CuO, Ni/kieselguhr, Ni-Cr oxide, Cu-Cr oxide, and 100Co/12.2MgO/260 kieselguhr) were studied in the gas phase hydroamination of the title compound (180-240 °C). In all cases piperidine, tetrahydrofurfurylamine, and their derivs. were the main products.

ACCESSION NUMBER: 1996:571680 CAPLUS
 DOCUMENT NUMBER: 126:18729
 TITLE: Reaction of tetrahydrofurfuryl alcohol with ammonia and hydrogen in the presence of heterogeneous catalysts
 AUTHOR(S): Glebov, L. S.; Kliger, G. A.; Shuikin, A. N.; Zaikin, V. G.
 CORPORATE SOURCE: Gubkin, I.M., Gosudarstvennaya Akademiya Nefti i Gaza, Moscow, Russia
 SOURCE: Neftekhimiya (1996), 36(4), 344-350
 CODEN: NEFTAH; ISSN: 0028-2421
 PUBLISHER: Nauka
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 80 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The inhibiting action of complexes of transition metals with macrocyclic polyamine-2,4-dioxo-1,5,8,12-tetraazatetradecane, containing the stearic acid group as a counterion (crown-complexes), on the initiated chain oxidation of cyclohexanol is investigated. The crown-complexes of Co(II) and Cu(II) are less efficient in the chain termination of oxidation than stearates of the same metals, whereas the Mn(II) activity depends on the temperature of preparation of the starting solution and approaches that of MnSt₂ stearate. The Zn(II) complex does not suppress alc. oxidation. Each mol. of the transition metal-complex is capable of catalyzing the rupture of more than 100 chains per h. The catalytic decay of the alc. peroxy radicals (ROO•) proceeds via alternating redox reactions involving Mn³⁺ and Mn(n+1)²⁺. The low values of the rate consts. for the interactions of ROO• with Co, Cu, and Mn, resp., are equal to: keff(1 mol-l s-l) .apprx.> 5 + 105, 1.4 + 106, and 5.4 + 107 at 343 K; 1.1 + 105, 1.2 + 106, and 4.3 + 107 at 363 K. The main reason for decreasing keff with a rise in temperature is an addnl. chain generation due to the reactions of Mn³⁺ and Mn(n+1)²⁺ with H₂O₂ accumulated in the course of cyclohexanol oxidation. The equation relating keff to [M]O, [H₂O₂], and the rate of alc. oxidation by mol. oxygen is presented.

ACCESSION NUMBER: 1996:74736 CAPLUS
 DOCUMENT NUMBER: 124:260170
 TITLE: Catalytic decay of cyclohexanol peroxy radicals on crown complexes of transition metals
 AUTHOR(S): Aleksandrov, A. L.; Marchenko, E. P.; Pokidova, T. S.
 CORPORATE SOURCE: Inst. Chemical Physics Chernogolovka, Russian Academy of Sciences, Moscow, 142432, Russia
 SOURCE: Kinetics and Catalysis (Translation of Kinetika i Kataliz) (1995), 36(6), 757-61
 CODEN: KICAAH; ISSN: 0023-1584
 PUBLISHER: MAIK Nauka/Interperiodica
 DOCUMENT TYPE: Journal
 LANGUAGE: English

L20 ANSWER 81 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The vapor-phase reaction of 1-pentanol with 2-ethylhexylamine and of 1-heptanol with cyclohexylamine over the industrial CuO-ZnO-Al₂O₃ catalyst SNM-1 was studied. The corresponding, asym. secondary and tertiary amines in yields of 59-99% and 5-49%, resp., were produced at 175-185 °C.

ACCESSION NUMBER: 1995:848208 CAPLUS
 DOCUMENT NUMBER: 124:55321
 TITLE: Synthesis of asymmetric secondary and tertiary amines from a primary amine and alcohol over the industrial catalyst for methanol synthesis SNM-1
 AUTHOR(S): Shuikin, A. N.; Glebov, L. S.; Kliger, G. A.; Zaikin, V. G.
 CORPORATE SOURCE: A.V. Topchiev Institute Petrochemical Synthesis, Russian Academy Sciences, Moscow, 117912, Russia
 SOURCE: Izvestiya Akademii Nauk, Seriya Khimicheskaya (1993), (4), 799-800
 CODEN: IASKEA
 PUBLISHER: Nauka
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian
 OTHER SOURCE(S): CASREACT 124:55321

L20 ANSWER 82 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB The reactions of alanine decyl ester (AlaODc) with nucleophiles were catalyzed by a crosslinked polystyrene-supported ethylenediamine-copper(II) ion complex (Cu en-PS) under toluene/resin two-phase conditions or toluene/aqueous buffer/resin three-phase conditions. In the two-phase reactions with octylamine, the rate was increased with a decreasing metal content of the resin. The catalytic efficiency was also dependent on other factors, i.e., metal ion, ligand structure, and substrate structure. In the three-phase reactions with water, the hydrolysis was very fast when acetate ions were present in the aqueous phase. A substrate-coordinated intermediate complex is suggested under both reaction conditions.

ACCESSION NUMBER: 1995:653282 CAPLUS

DOCUMENT NUMBER: 123:340790

TITLE: Reactions of amino acid decyl esters with nucleophiles

catalyzed by polymer-supported amine-metal

complexes

AUTHOR(S): Ohtani, Noritaka; Inoue, Yukihiko; Inagaki, Yuichi;

Fukuda, Kenji; Nishiyama, Taisuke

CORPORATE SOURCE: Dep. Materials Eng. and Applied Chemistry, Akita

Univ., Akita, 010, Japan

SOURCE: Bulletin of the Chemical Society of Japan (1995),

68(6), 1669-75

CODEN: BCSJAB; ISSN: 0009-2673

PUBLISHER: Nippon Kagakki

DOCUMENT TYPE: Journal

LANGUAGE: English

L20 ANSWER 84 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB Coating materials contain cationic resins prepared by the reaction of epoxy resins with compds. having OH, secondary amino, and amide groups, and compds. having primary OH and primary or secondary amino groups, epoxides having alicyclic and/or bridged alicyclic skeletons, and hydroxides and organic acid salts (except Pb salts) of elements of atomic number 25-30 or 40-42.

Thus, a coating material contained a cationic resin prepared from EHPE 3150 (I), diethanolamine, stearic acid-hydroxyethylaminoethylamine reaction product, bisphenol A, and bisphenol A diglycidyl ether, a I hardening agent, Cu(OH)2, and a pigment paste.

ACCESSION NUMBER: 1994:325858 CAPLUS

DOCUMENT NUMBER: 120:325858

TITLE: Cationic electrodeposition coating materials

INVENTOR(S): Katayama, Teiji; Kasukawa, Takahisa; Ichinose,

Hiroshi; Fujibayashi, Toshio

PATENT ASSIGNEE(S): Kansai Paint Co Ltd, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 18 pp.

CODEN: JKOXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06033001	A2	19940208	JP 1992-210875	19920715
PRIORITY APPLN. INFO.:			JP 1992-210875	19920715

L20 ANSWER 83 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB A Dynamic (Pulse)-In-situ FT-IR Technique has been developed, by which the wave nos. of the intermediates on the catalyst surface can be measured and recorded during the reaction. According to the appearance sequence of these waves, their peak areas and the time of their formation, the reaction mechanism may be studied more directly and precisely than the traditional methods. By correlating the peak areas and the concns. of the surface intermediates, we could study the complex reaction network involving the surface intermediates and obtain the corresponding kinetic parameters, and in addition, determine the rate controlling step. Based on

the Pulse-in-situ technique, we have also developed a "Chemical Trapping" approach to examine and determine the kinds of the intermediates on the catalyst surface in low-carbon alc. production and study the mechanism of the forced periodic controlling operation.

ACCESSION NUMBER: 1995:953 CAPLUS

DOCUMENT NUMBER: 122:12446

TITLE: Development of the dynamic (pulse)-in situ technique

and its applications in the study of heterogeneous

catalysis

AUTHOR(S): Yuan, Naiju; Hu, Jianli; Ding, Fuxin; Zhu, Qiming; Li,

Jinlu

CORPORATE SOURCE: Dep. Chem. Eng., Tsinghua Univ., Beijing, 100084,

Peop. Rep. China

SOURCE: Huaxue Fanying Gongcheng Yu Gongyi (1993), 9(1),

98-107

CODEN: HFGGEU; ISSN: 1001-7631

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

L20 ANSWER 85 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN

AB Aliphatic tert-amines R3R4NR5 (R3 = C8-36 alkyl or alkenyl; R4, R5 = C1-3 alkyl, C8-36 alkyl or alkenyl), which are prepared by heating C8-26

aliphatic

alcs. with NH3 or aliphatic primary or secondary amines R1NHR2 (R1 =

H, C1-3 alkyl; R2 C1-3 alkyl) in the presence of a copper

catalyst Cu-based hydrogenation or dehydrogenation

catalyst, are contacted with pressurized H at H 6-100, H2O

50.2, and raw material amines 50.1 atm partial pressure and

80-280° and then the Cu-based catalyst is separated

In a continuous process of the aliphatic tert-amines, the gaseous part and

the liquid part are separated before treating the amines with pressurized H

at the specified temperature This process markedly suppresses the

contamination of

the Cu-based catalyst into the product amines and

provides the aliphatic tert-amines with excellent quality stability and very

little elution of Cu and without heating coloration. Thus,

dodecyl alc. containing 1% Cu-chromite at 800 g/h and MeNH

at 1.7-2.6 mol/h were fed into a mixture of 2.5 kg

monomethyldodecylamine (I) and 50 g Cu-chromite at

hydrogen partial pressure 10 and 230° while circulating the gas at

150 L/h to give a product. The product effluent was cooled to 120°

and after separating H(g) containing H2O and MeNH and a crude liquid

containing the

catalyst and I, the latter liquid was continuously fed at .apprx.800

g/h into a pressurized hydrogen-treatment bath at H pressure 10 atm and

200° to give, after separation of the catalyst, 97.6% I of

98.5% purity containing 0.05 ppm Cu with heat coloration hue (APHA

value) 10.

ACCESSION NUMBER: 1994:216702 CAPLUS

DOCUMENT NUMBER: 120:216702

TITLE: Preparation of aliphatic tertiary amines

INVENTOR(S): Okajima, Naoyoshi; Nakazawa, Mikiro

PATENT ASSIGNEE(S): Shin Nippon Rika KK, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.

CODEN: JKOXAF

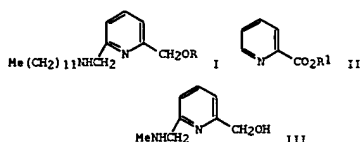
DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05301846	A2	19931116	JP 1991-96224	19910401
JP 3003256	B2	20000124		
PRIORITY APPLN. INFO.:			JP 1991-96224	19910401
OTHER SOURCE(S):			MARPAT 120:216702	



AB Micellar aggregates of complexes of transition metal ions with the hydroxy-functionalized surfactant I (R = H) are very effective catalysts of the cleavage of activated esters of α -amino acids. To ascertain their effectiveness toward unactivated esters, a systematic kinetic study was undertaken employing as substrates the picolinic acid esters II (R₁ = (unsubstituted Ph, Et, trifluoroethyl, CH₂CH₂OMe), the pK_a of their alc. portion spanning >12 units from 3.6 to 16. The leaving group effect was investigated in water, pH = 6.3, in the absence and presence of Cu²⁺ ions, in the presence of the nonmicellar complex III·Cu²⁺, and in the presence of micellar aggregates made of I (R = H, Me)·Cu²⁺. In the presence of free metal ions the leaving group effect is negligible in the case of esters with good leaving groups (pK_a < ca. 12), and it becomes remarkably large in that of unactivated substrates. In the presence of Cu²⁺ complexes, either micellar I (R = H) or nonmicellar III, the leaving group effect is relatively small in the case of activated substrates [pK_a < 9, I (R = H); pK_a < 11, III] and sharply increases in the case of unactivated substrates. A similar trend was observed in a less extensive kinetic investigation using Zn²⁺ ions at pH = 7.5. The largest rate enhancements were observed in the case of the most activated substrates in micellar

sols.

of the I (R = H)·Cu²⁺ complex [1.6 + 10⁶ fold for II (R₁ = 2,4-dinitrophenyl) over the rate in pure buffer], considerably larger than those in the presence of its nonmicellar analog (4.2 + 10⁴ fold) or of the free metal ion (1.5 + 10³ fold). However, in the case of unactivated esters, such kinetic benefits vanish and the metal ion alone is even more effective [2 + 10⁴ fold acceleration for II (R₁ = Et) in the presence of Cu²⁺] than its complexes, either in the monomeric (7.3 + 10³ fold) or in the micellar form (4.6 + 10³ fold). On the basis of possible changes in the mechanistic pathway, depending on the nature of the leaving group, a rationale is offered.

ACCESSION NUMBER: 1994:216343 CAPLUS
DOCUMENT NUMBER: 120:216343
TITLE: Leaving group effect in the cleavage of picolinate esters catalyzed by hydroxy-functionalized metallomicelles
AUTHOR(S): Scrimin, Paolo; Tacilla, Paolo; Tonellato, Umberto
CORPORATE SOURCE: Dep. Org. Chem., Univ. Padova, Padova, 35131, Italy
SOURCE: Journal of Organic Chemistry (1994), 59(1), 18-24
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal

L20 ANSWER 87 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A method for manufacturing a Ni alloy catalyst involves alloying, in an alc. atmospheric, catalytic components of Ni, a rare earth element, and 21 of Cu, Re, Ir, Pt, Pd, Cr, Mo, Mn, Co, Rh, Ag, Au, Zn, Fe, and Ru. A method for manufacturing an ethyleneamine involves reacting NH₃ and/or ethyleneamine with ethanolamine in the presence of the above catalyst and H₂ to form an ethyleneamine having an increased ethylene chain number

ACCESSION NUMBER: 1994:174611 CAPLUS
DOCUMENT NUMBER: 120:174611
TITLE: Manufacture of nickel alloy catalyst and ethyleneamine
INVENTOR(S): Hironaka, Toshio; Nagasaki, Nobutaka; Hara, Yasushi
PATENT ASSIGNEE(S): Tosoh Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 12 pp.
CODEN: JKKKAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

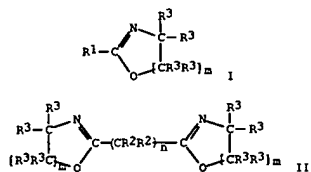
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05168934	A2	19930702	JP 1991-356873	19911226
JP 3265508	B2	20020311	JP 1991-356873	19911226

PRIORITY APPLN. INFO.:

L20 ANSWER 88 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Vapor-phase N-alkylation of cyclohexylamine and 2-ethylhexylamine with 1-heptanol and 1-pentanol was carried out in the presence of H₂ on the industrial SNM 1 catalyst, CuO (52-54%)-ZnO (24-28%)-Al₂O₃ (5-6%), and on the catalyst containing Fe₃O₄, CuO, and V₂O₅. The highest yields of secondary and tertiary amines (99% total yield) were obtained at 175-195° and amine-alc. ratio 0.2.

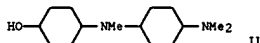
ACCESSION NUMBER: 1994:79876 CAPLUS
DOCUMENT NUMBER: 120:79876
TITLE: Catalytic N-alkylation of primary amines by alcohols
AUTHOR(S): Shuikin, A. N.; Glebov, L. S.; Kliger, G. A.; Zaikin, V. G.
CORPORATE SOURCE: Inst. Neftekhim. Sint. im. Topchieva, Moscow, Russia
SOURCE: Neftekhimiya (1993), 33(4), 321-6
CODEN: NEFTAH; ISSN: 0028-2421
DOCUMENT TYPE: Journal
LANGUAGE: Russian



AB The title compds. [I, II; R1 = C1-36 linear or branched alkyl or alkenyl, C1-15 linear or branched alkyl-(un)substituted Ph; R2 = H, C1-36 linear or branched alkyl or alkenyl, C1-15 linear or branched alkyl-(un)substituted Ph; R3 = H, C1-36 linear or branched alkyl or alkenyl; m = 1, 2; n = 1-19], useful as intermediates for poly(N-acyl)ethylene imines which are suitable for cleaning agents and emulsifying agents, are prepared by cyclocondensation of R1CN (II; R1 = same as above) or NC(CR2R2)nCN (III; R2, n = same as above) with HO(CR3R3)(CR3R3)mNH2 (IV; R3, m = same as above) with removal of NH3 formed, in the presence of at least one catalyst selected from oxides, sulfates, halides, and carboxylates of Li, Al, Ca, Mn, Mg, Cu, Zn, Cd, Co, Fe, and Ni under the condition of (a) 0.1-10 mol ratio of III/(I or II) and maintaining the H2O content of the system 50.5 weight% or (b) 1.5-10 mol ratio of III/(I or II) and maintaining the H2O content of the system 0.5-1.0 weight%. The process gives I and II of high purity in high yields. Thus, MeCN 82.1, H2NCH2CH2OH 244, and Zn(OAc)2 (dehydrated at 105° for 10 h) 9.17 g were charged in a reactor and heated at 85° under the flow of dry N for 20 h to give, after distillation, 90% 2-methyl-2-oxazoline of 99.5% purity vs. 65 and 99.5%, resp., for using undried Zn(OAc)2·2H2O.

ACCESSION NUMBER: 1993:603399 CAPLUS
DOCUMENT NUMBER: 119:203399
TITLE: Preparation of cyclic imino ethers by cyclocondensation of nitriles with amino alcohols
INVENTOR(S): Tanaka, Toshinori; Matsunaga, Akira; Nagumo, Hiroshi; Oda, Taku
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05140129	A2	19930608	JP 1991-300557	19911115
PRIORITY APPL. INFO.:			JP 1991-300557	19911115
OTHER SOURCE(S):			CASREACT 119:203399; MARPAT 119:203399	



AB HO(RN(R1))nR1 [R = C2-24 linear or branched alkyl alkylene, aliphatic alkylene, cycloalkylene, arylene, aralkylene, (CH2CH2O)p(CH2CH2)q; p = 0, pos.; integer; q = pos. integer; R1 = C1-24 linear or branched alkyl, aryl, aralkyl; n = 2-50] are prepared by reaction of a diol or an aldehyde with a primary amine followed by amination with a secondary amine in the presence of a Cu - 4-th period transition metal element - group VIII Pt group element catalyst optionally comprising an alkali metal or alkaline earth metal. The 4-th period transition metal element is selected from Cr, Mn, Fe, Co, Ni, and Zn and the group VIII Pt group element is selected from Pt, Pd, Ru, and Rh. Thus, 10% aqueous Na2CO3 was slowly added dropwise to a stirred mixture of a synthetic zeolite (carrier) and an aqueous solution of Cu (NO3)2, Ni(NO3)2, and PdCl2 (Cu:Ni:Pt metal mol ratio = 4:1:0.1) at 90°; after ripening for 1 h, a precipitate was filtered, washed with H2O, dried at 80° for 10 h, and fired at 400° for 1 h to give a catalyst. This catalyst (24 g) and 600 g 1,6-hexanediol were heated with stirring when the temperature reached 100°, H was blown into the mixture at 10 L/h.kg while raising the temperature to 200°; a gaseous mixture of MeNH2 and H was blown into the reaction mixture at 200° and 60 L/h.kg for .apprx.4 h to give a viscous compound. A gaseous mixture of Me2NH and H was blown into the latter product in the presence of the same catalyst at 160°; the catalyst was removed by filtration to give a light yellow liquid which was distilled at 210-220° (temperature at the top of a distillation column) and 0.3 torr to give 40.3% a tert-amine (II).

ACCESSION NUMBER: 1993:580449 CAPLUS
DOCUMENT NUMBER: 119:180449
TITLE: Preparation of tertiary amino alcohol having terminal hydroxy group by catalytic amination of diol or aldehyde with primary amine
INVENTOR(S): Sotodani, Koshiro; Abe, Yutaka; Fukushima, Tetsuro
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05140051	A2	19930608	JP 1991-307681	19911122
JP 2941524	B2	19990825		
PRIORITY APPL. INFO.:			JP 1991-307681	19911122
OTHER SOURCE(S):			CASREACT 119:180449; MARPAT 119:180449	

AB Hair cosmetics contain (R1N+R2R3R4)X- (R1 = branched C8-28 alkyl; R2 = straight-chain C8-22 alkyl or alkenyl; R3, R4 = C1-4 alkyl, H; R3 = R4 = H; X = halo, C1-4 alkyl sulfate) and R6(OCH2CHR5)2OH (R5 = H, Me; R6 = C1-5 alkyl). The cosmetics show hair-softening and -smoothing effects. Fine Oxocool 180N [2-(3-methylhexyl)-7-methyl-1-decanol] was treated with Cu-Zn-Ru catalyst (preparation given) and MeNH2 gas at 190° to give 93% N-[2-(3-methylhexyl)-7-methyl-1-decyl]-N-methylamine, which was treated similarly with Kalcobl 20 (n-dodecyl alc.) for .apprx.6 h to give 90% N-[2-(3-methylhexyl)-7-methyl-1-decyl]-N-dodecyl-N-methylamine (I). I, iso-Pr alc., Na2CO3, H2O, and MeCl were kept at 100° for .apprx.8 h to give 71% N-[2-(3-methylhexyl)-7-methyl-1-decyl]-N-dodecyl-N,N-dimethylammonium chloride (II). A hair rinse composition containing II, stearyltrimethylammonium chloride, diethylene glycol monobutyl ether, cetyl alc., propylene glycol, and H2O was formulated.

ACCESSION NUMBER: 1993:524827 CAPLUS
DOCUMENT NUMBER: 119:124827
TITLE: Hair cosmetics containing quaternary ammonium salts and/or amine salts and dialkylene glycol monoalkyl ethers
INVENTOR(S): Horinishi, Nobutaka; Yahagi, Kazuyuki
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05097632	A2	19930420	JP 1991-253831	19911001
JP 3043486	B2	20000522		
PRIORITY APPL. INFO.:			JP 1991-253831	19911001
OTHER SOURCE(S):			MARPAT 119:124827	

L20 ANSWER 92 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Carbamates are prepared by oxidative carbonylation of primary or secondary amines or ureas with CO in presence of an alc., an O-containing oxidizing agent, metalloporphyrin or metal phthalocyanine catalyst derived from Group IIIa-Va and Group VIII metals, and an iodine-containing promoter. Decomposition of carbamates prepared in this manner affords isocyanates. Thus, reaction of 3.0 g tert-BUNH₂, 0.20 g CoPc (Pc = phthalocyanine dianion), and 1.0 g NaI with 40 g EtOH under 80 psi O₂/1520 psi CO afforded 99% yield of Et N-tert-Bu carbamate.

ACCESSION NUMBER: 1993:494707 CAPLUS
DOCUMENT NUMBER: 119:94707
TITLE: Processes for producing carbamates and isocyanates
INVENTOR(S): Leung, Tak W.; Dombek, Bernard D.
PATENT ASSIGNEE(S): Union Carbide Chemicals and Plastics Technology Corp., USA
SOURCE: U.S., 13 pp.
CODEN: USXKAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5194660	A	19930316	US 1990-631962	19901221
PRIORITY APPL. INFO.:			US 1990-631962	19901221
OTHER SOURCE(S):		CASREACT 119:94707		

L20 ANSWER 93 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

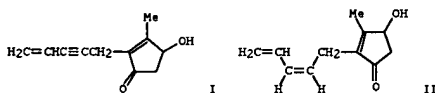
AB Hair cosmetics contain [R1NR2R3R4]X- (R1 = straight-chain saturated or branched C8-28 alkyl; R2 = C8-22 alkyl or alkenyl; R3, R4 = C1-4 alkyl, H; R3 = R4 = H; X = halo, C1-4 alkyl sulfate) and silicones chosen from di-Me polysiloxane, Me Ph polysiloxane, amino-modified silicone, fatty acid-modified polysiloxane, alc.-modified silicone, aliphatic alc.-modified polysiloxane, polyether-modified silicone, epoxy-modified silicone, F-modified silicone, cyclosilicone, and alkyl-modified silicone. The cosmetics show hair-smoothing and -softening effects. Fine Oxocool 180N [2-(3-methylhexyl)-7-methyl-1-decanol] was treated with Cu-Zn-Pu catalyst (preparation given) and MeNH₂ gas at 190° to give 93% N-[2-(3-methylhexyl)-7-methyl-1-decyl]-N-methylamine, which was treated similarly with Kalcobl 20 (n-dodecyl alc.) for .apprx. 6 h to give 90% N-[2-(3-methylhexyl)-7-methyl-1-decyl]-N-dodecyl-N-methylamine (II). 1, iso-Pr alc., Na₂CO₃, H₂O, and MeCl were kept at 100° for .apprx. 8 h to give 71% N-[2-(3-methylhexyl)-7-methyl-1-decyl]-N-dodecyl-N,N-dimethylammonium chloride (II). Hair rinse composition containing II, stearyltrimethylammonium chloride, polyether-modified silicone,

catyl alc., propylene glycol, and H₂O was formulated.
ACCESSION NUMBER: 1993:479819 CAPLUS
DOCUMENT NUMBER: 119:79819
TITLE: Hair cosmetics containing quaternary ammonium salts and/or amine salts and silicones
INVENTOR(S): Horinishi, Nobutaka; Yahagi, Kazuyuki
PATENT ASSIGNEE(S): Kao Corp., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 15 pp.
CODEN: JKKCAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05070327	A2	19930323	JP 1991-230253	19910910
JP 3043481	B2	20000522		
PRIORITY APPL. INFO.:			JP 1991-230253	19910910
OTHER SOURCE(S):		MARPAT 119:79819		

L20 ANSWER 94 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

GI



AB The title intermediate I is prepared by reacting a propargylcyclopentenone with vinyl halide in presence of a Pd catalyst, Cu catalyst and an amine. I is reduced with Zn in aqueous alc. to give the title pyrethrolone (II) which is an intermediate for production of insecticidal pyrethrins. (+)-(S)-2-Methyl-3-propargylcyclopent-2-en-4-on-1-ol in C₆H₆ was added to Pd(PPh₃)₄, CuI and Et₃N to give (+)-(S)-I which under N atmospheric was added to aqueous 0.1 N HCl and Zn powder to give (+)-(S)-II.

ACCESSION NUMBER: 1993:255149 CAPLUS
DOCUMENT NUMBER: 118:255149
TITLE: Process for producing pyrethrolone and its intermediate compound
INVENTOR(S): Takagaki, Toshi; Matsuo, Noritada
PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan
SOURCE: Eur. Pat. Appl., 7 pp.
CODEN: EPXKDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 521449	A2	19930107	EP 1992-111034	19920630
EP 521449	A3	19930414		
EP 521449	B1	19961106		
R: CH, DE, FR, GB, IT, LI, NL				
JP 05009148	A2	19930119	JP 1991-160471	19910701
JP 3057816	B2	20000704		
CA 2068966	AA	19930102	CA 1992-2068966	19920519
US 5225605	A	19930706	US 1992-897064	19920611
PRIORITY APPL. INFO.:			JP 1991-160471	A 19910701

L20 ANSWER 95 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB HO[2n(2n+1)R₁R₂]nZOH (1; R₁, R₂ = C1-4 alkyl; Z = C2-24 alkylene, C3-24 cycloalkylene, C6-24 arylene, (CH₂CH₂O)_p(CH₂CH₂q etc.; Z1 = C1-9 alkylene; n = 1-50; p, q ≥ 0), useful as catalytically active polyol components in producing polyurethane (foams), were prepared by reacting a diol or dialdehyde with a diamine, in the presence of a catalyst containing Cu, a Group IV transition metal, a Group VIII metal, and optionally an alkali or alkaline earth metal. Thus, the

title amino alc. 1 (R₁ = R₂ = Me, Z = C₆H₁₂, Z1 = C₃H₆, n = 2.7) (II) was prepared by condensation reaction of 1,6-hexanediol with Me₂NCH₂CH₂CH₂NMe₂ over 5 h at 200° in the presence of a Cu /Ni/Pd catalyst. A formulation comprising II, a mixture of aromatic amine-, sucrose polyether-, and glycerol polyether polyols, polyisocyanate TR-50EX, glycerol, foam stabilizer L-5340, H₂O, and foaming agent R-123 gave a rigid polyurethane foam with a better mold filling than a similar mixture containing Me₂N(CH₂)₆NMe₂ as a catalyst and no I.

ACCESSION NUMBER: 1993:148717 CAPLUS
DOCUMENT NUMBER: 118:148717
TITLE: Preparation of novel tertiary amino alcohols as catalytically active diol components for polyurethane foams with improved moldability
INVENTOR(S): Abe, Hiroshi; Fukushima, Tetsuaki; Sotoya, Kohshiro; Harada, Shoichiro; Kitagawa, Hiroshi; Morii, Masayoshi; Isayama, Yasutoshi
PATENT ASSIGNEE(S): Kao Corp., Japan
SOURCE: Eur. Pat. Appl., 34 pp.
CODEN: EPXKDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 502516	A2	19920909	EP 1992-103760	19920305
EP 502516	A3	19930310		
R: DE, ES, FR, GB				
JP 04279554	A2	19921005	JP 1991-38574	19910305
JP 2895973	B2	19990531		
JP 05093031	A2	19930416	JP 1992-32014	19920219
JP 3221712	B2	20011022		
CA 2062269	AA	19920906	CA 1992-2062269	19920304
US 5315041	A	19940524	US 1992-845594	19920304
US 5440099	A	19950822	US 1993-123165	19930920
PRIORITY APPL. INFO.:			JP 1991-38574	A 19910305
			JP 1991-43849	A 19910308
			JP 1992-32014	A 19920219
			US 1992-845594	A3 19920304

L20 ANSWER 96 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB In preparation of the title compds. R3NR1R2 (R1, R2 = C8-36 alkyl or alkenyl);
 R3 = C1-3 alkyl, C8-36 alkyl or alkenyl) by thermal dehydration reaction of C8-36 aliphatic alcs. with NH3 or C1-3 aliphatic primary amines under H₂ Cu catalysts containing 21 Cu -chromite, Cu Zn oxide, Cu Mo oxide, Cu V oxide, Cu Ni oxide, Cu Cr Zn oxide, Cu oxide, and their Ba-, Mg-, or modified compds., and Raney Cu are used, at H pressure 6-100 atm, and partial NH3 or C1-3 aliphatic primary amine pressure 50.1 atmospheric An autoclave containing dodecyl alc. and Cu-chromite was fed with MeNH2 at 0.005-0.05 atm under 10 atm H₂ at 220° for 2.5 h to give 96.2% monomethyldidodecylamine with APHA 10, which was quaternized by MeCl at 100° for 3 h to give a tetraalkylammonium-type cationic surfactant.

ACCESSION NUMBER: 1993:83321 CAPLUS
 DOCUMENT NUMBER: 118:83321
 TITLE: Preparation of aliphatic tertiary amines for surfactants
 INVENTOR(S): Okajima, Naoyoshi; Nakazawa, Mikiro; Ogawa, Yasuaki
 PATENT ASSIGNEE(S): New Japan Chemical Co., Ltd., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
 CODEN: JKKXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 04230246	A2	19920819	JP 1991-106877	19910410
JP 3127480	B2	20010122		
KR 211777	B1	19990802	KR 1991-16574	19910920
			JP 1990-253420	A1 19900920

PRIORITY APPLN. INFO.:
 OTHER SOURCE(S): MARPAT 118:83321

L20 ANSWER 97 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The title polymers are prepared by using a catalyst composition containing radical sources and metal compds. Thus, heating ethylene glycol monoacetate 229, Ce(NH4)2(NO3)6 32.9, and Bu acrylate 128 parts in EtOH at 53° for 8 h gave 100% polymers having number-average mol. weight 5100, and terminal functionality 1.3.

ACCESSION NUMBER: 1993:81618 CAPLUS
 DOCUMENT NUMBER: 118:81618
 TITLE: Preparation of polymers bearing terminal functional groups
 INVENTOR(S): Matsunaga, Toshiaki; Yoshida, Masatoshi; Namura, Ichiro; Takei, Kazuo; Tamura, Fumihide
 PATENT ASSIGNEE(S): Nippon Shokubai Co., Ltd., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 13 pp.
 CODEN: JKKXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 04202504	A2	19920723	JP 1990-339708	19901130
			JP 1990-339708	19901130

PRIORITY APPLN. INFO.:

L20 ANSWER 98 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB N-Alkyl-N-methylamines and N-alkenyl-N-methylamines are prepared in high yield by reaction of higher alcs. with MeNH2 in the presence of catalysts containing Cu, another 4th-period transition element (except Cr), and optionally a Pt-group element, at 1-100 atm and 100-250°, with both introduction of H2 and removal of formed H2O, and with the exhaust gas regulated to contain 5-50% MeNH2 by volume For example, to 1200 g stearyl alc. and 6 g Cu-Ni catalyst (on zeolite, calcined to oxide form) were fed H2 at 40 L/h and 100-190°, followed by introduction of MeNH2 (to form 20% of 'exhaust gases) at 190° and atmospheric pressure. N-Stearyl-N-methylamine was obtained in 85% yield, vs. only 51% for a com. Cu-Cr catalyst. Addnl. alcs. such as (C7H15) (C9H19)CHCH2OH (Diadol 18G), 2-(3-methylhexyl)-7-methyl-1-decanol (Fine Oxocohol 180N), Dobanol 23-I, and lauryl alc. (Kalcobl 20) gave corresponding N-Me amines in yields of 84-94%, using Cu-Ni, Cu-Zn, Cu-Zn-Ru, Cu-Co-Pd, Cu-Ni-Pt, and Cu-Zn-Pd catalysts on zeolites, titania, alumina, or diatomaceous earth supports.

ACCESSION NUMBER: 1993:59282 CAPLUS
 DOCUMENT NUMBER: 118:59282
 TITLE: Process for producing N-alkyl-N-methylamines or N-alkenyl-N-methylamines from alcohols and methylamine
 INVENTOR(S): Abe, Hiroshi; Taniguchi, Hideaki; Nishimoto, Yoshifumi; Setoya, Kohshiro
 PATENT ASSIGNEE(S): Kao Corp., Japan
 SOURCE: Eur. Pat. Appl., 8 pp.
 CODEN: EPXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 510493	A2	19921028	EP 1992-106429	19920414
EP 510493	A3	19940406		
EP 510493	B1	19960221		
R: DE, ES, FR, GB				
JP 05112495	A2	19930507	JP 1992-80778	19920402
JP 2706595	B2	19980128		
ES 2083016	T3	19960401	ES 1992-106429	19920414
US 5296631	A	19940322	US 1992-871541	19920421
			JP 1991-90270	A 19910422

PRIORITY APPLN. INFO.:
 OTHER SOURCE(S): CASREACT 118:59282

L20 ANSWER 99 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Conjugates of redox cofactors and cyclodextrins that have the catalytic and kinetic properties of redox enzymes are synthesized. These catalysts are phys. and chemical more stable than enzymes catalyzing the same reaction. The synthesis of flavin conjugates with cyclodextrin is by conjugation of a 6-iodo-cyclodextrin with o-phenylenediamine or a derivative, e.g. a nitroaniline, that is then conjugated with an alloran derivative to generate the flavin moiety. This synthetic method is much more efficient than those of the prior art. 4-Methylamino-3-nitro-benzaldehyde (I) was prepared by refluxing an ethanolic solution of 4-chloro-3-nitrobenzaldehyde and methylamine. I was reduced to the alc. by reduction with NaBH4 and this converted to 4-methylamino-3-nitrobenzyl chloride (II) by reaction with SOCl2. II was conjugated with β -cyclodextrin in the presence of NaH to give 2-O-(4-methylamino-3-nitro)benzyl- β -cyclodextrin that was then hydrogenated and condensed with alloran to give mono-2-flavo- β -cyclodextrin (III). III catalyzed the oxidation of dihydronaphthylnicotinamide with Michaelis-Menten kinetics (kcat=2.82 \times 10⁻²s⁻¹; Ka=277 M⁻¹).

ACCESSION NUMBER: 1993:55130 CAPLUS
 DOCUMENT NUMBER: 118:55130
 TITLE: Artificial redox enzymes using redox coenzymes conjugated to cyclodextrins
 INVENTOR(S): D'Souza, Valerian T.; Rong, Ding
 PATENT ASSIGNEE(S): University of Missouri, USA
 SOURCE: PCT Int. Appl., 67 pp.
 CODEN: PIXX02
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9217508	A1	19921015	WO 1992-US2608	19920331
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, MC, NL, SE				
US 5258370	A	19931102	US 1991-680511	19910404
			US 1991-680511	A 19910404

PRIORITY APPLN. INFO.:

L20 ANSWER 100 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Aliphatic amines are prepared by a 2-stage amination of C2-9 fatty alcs., over $XaYbZcd$ ($X = Cu, Co, Ni, Y = Cr, Mo, W, Z = Al, Si, a = 1, b = 0, 0.01-0.8; c = 1-5; d = a$ value to satisfy valence) and $AVbWcZdYz$ ($A = Co, Ni, Pt, Pd, Ni, B = Zn, Cd, Cu, Mg, Ca, Mn; C = Cr, V; D = Al, Si; V = 1, W = 0.1-2; x = 0, 0.1-1.5; y = 1-15; z = a$ value to satisfy valence) catalysts, resp. A mixture of 2-ethylhexanol, H, and NH_3 was passed over $CuSi1.503$ at 260° and atmospheric pressure for 120 h and distilled to give a mixture of isooctylamine (I) 8.4, bis(2-ethylhexyl)amine (II) 25.9, isooctyl nitrile 59.5, isooctanol 2.1, and tris(2-ethylhexyl)amine 0.5 weight%, which was passed over $CoZnSi7.508.5$ at 200° and atmospheric pressure to give I 89.5, II 7.8, isooctanol 1.5, and isooctyl nitrile 0.3 weight%.

ACCESSION NUMBER: 1992:447914 CAPLUS

DOCUMENT NUMBER: 117:47914

TITLE: preparation of aliphatic amines from C2-9 fatty alcohols

INVENTOR(S): Xie, Jiaming; Cheng, Yongtai

PATENT ASSIGNEE(S): China Petrochemical Engineering Co., Peop. Rep. China

SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 17 pp.

CODEN: CNDXEV

DOCUMENT TYPE: Patent

LANGUAGE: Chinese

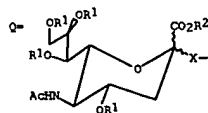
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CN 1057831	A	19920115	CN 1990-102887	19900630
CN 1025732	B	19940824		
PRIORITY APPLN. INFO.:			CN 1990-102887	19900630

L20 ANSWER 101 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

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AB Title glycolipid derivs. $Q(CH_2)nCH_2(CH_2)lA$ [$l: R1 = H, Ac; R2 = H, C1-4$ alkyl, alkali metal ion, alkaline earth metal ion, ammonium ion; $X = O, S, O(CH_2)mCH_2CO, O(CH_2)mCONH_2; m = 1-10; n, l = 0-3; A = H, C10-40$ straight chain or branched acylamino, alkyl, alkenyl, alkoxy, alkenyloxy, alkylthio, alkenylthio; $B = H, CO_2H, (N-alkyl)carbamoyl, C10-30$ alkyl, alkenyl, alkoxy, alkenyloxy, acylamino, Q] are prepared by (1) reaction of QAc ($R1 = Ac, R2 = Me, X = O$) with $HX(CH_2)mCH_2(CH_2)lA$ (III) in the presence of a Lewis acid or (2) reaction of $QX1$ ($R1 = Ac, R2 = Me, X =$ single bond, $X1 = halo$) with III in the presence of a Lewis acid or its combination with a trityl halide. I are useful as components of liposome drug-delivery systems with good circulation life and reduced uptake by the reticuloendothelial system. Thus, a mixture of 400 mg mol. sieve 4A and 290 mg $ZnBr_2$ in CH_2Cl_2 was stirred 2.5 h at room temperature and thereto a mixture of

$\alpha-QX1$ ($R1 = Ac, R2 = Me, X =$ single bond, $X1 = halo$) 250, hexadecyl mercaptan 380, and mol. sieve 4A 150 mg in CH_2Cl_2 which was prestirred 2.5 h at room temperature, was added and the mixture was stirred 3 days at room temperature

to give 26% $\alpha-QX1$ ($R1 = Ac, R2 = Me, X1 = n$ -hexadecylthio) and 35% β -glycoside which were deacetylated with $MeONa$ in $MeOH$ and then saponified with 0.1N aqueous $NaOH$ in $MeOH$ to give $\alpha-QX1$ ($R1 = H, R2 = Na, X1 = n$ -hexadecylthio) (IV) and β -glycoside. A liposome suspension prepared from L- α -dipalmitoylphosphatidylcholine, cholesterol, [SH]inulin, and IV showed approx. twice the serum concentration than the control

liposome using dicetyl phosphate instead of IV in rats over 24 h.

ACCESSION NUMBER: 1992:59905 CAPLUS

DOCUMENT NUMBER: 116:59905

TITLE: Preparation of glycolipids containing sialic acid as pharmaceutical carriers

INVENTOR(S): Nakabayashi, Satoru; Higashi, Kunio; Miyoshi, Shiro; Yamauchi, Hitoshi

PATENT ASSIGNEE(S): Drug Delivery System Institute, Ltd., Japan

SOURCE: PCT Int. Appl., 175 pp.

CODEN: PIXX02

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9113079	A1	19910905	WO 1991-JP238	19910225
W: CA, JP, US				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, NL, SE				

L20 ANSWER 101 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CA 2050484	AA	19910828	CA 1991-2050484	19910225
EP 489162	A1	19920610	EP 1991-904345	19910225
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE				
JP 2563712	B2	19961218	JP 1991-504286	19910225
US 5243035	A	19930907	US 1991-752604	19910815
PRIORITY APPLN. INFO.:			JP 1990-46602	A 19900227
			JP 1990-75928	A 19900326
			JP 1990-75929	A 19900326
			JP 1990-166473	A 19900625
			WO 1991-JP238	W 19910225

OTHER SOURCE(S): MARPAT 116:59905

L20 ANSWER 102 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A nitro-alc. electrochem. reduction process for the industrial production of amino-alc. is described. The reaction takes place according to the scheme: $RNO_2 \rightarrow RNHOH \rightarrow RNH_2$. The hydroxylamine forms on a copper cathode on which a zinc deposit forms during electrolysis and this catalyzes the formation of the amine. Electroelectrodialysis is used to sep. the amino-alc. obtained as a concentrated solution from the sulfuric acid. The electrolysis cell is coupled with a recycling tank and production is discontinuous. The model proposed integrates in the cathodic process: the consecutive electrochem. reactions described above, electrodeposition of zinc and evolution of hydrogen. The calcn. takes into account the limitation of the reactions by the mass transfer together with the increase in volume of the catholyte during electrolysis. The model enables anticipation of the variation in the concentration of the various species

with time and the duration of the electrolysis in the galvanostatic mode; it also enables assessment of thermal release due to irreversibilities. The influence of operating conditions such as initial reagent concentration, programming c.d., ratio of the electrode surface with reagent quantity and electrolyte flow is presented. It is shown that for the operating conditions used in practice quasi-full conversion of the amino-alc. may be achieved without reaching the stage where hydroxylamine is exhausted as characterized by the limitation through mass transfer.

ACCESSION NUMBER: 1991:568946 CAPLUS

DOCUMENT NUMBER: 115:168946

TITLE: Amino-alcohol electrosynthesis. Modeling of a set-up for producing 2-amino-2-methyl-1,3-2 propanediol

AUTHOR(S): Savall, A.; Quesado, J.; Rignon, M.; Malafosse, J.

CORPORATE SOURCE: Lab. Genie Chim. Electrochim., Univ. Paul Sabatier, Toulouse, 31 062, Fr.

SOURCE: Journal of Applied Electrochemistry (1991), 21(9), 805-15

CODEN: JAELEJ; ISSN: 0021-891X

DOCUMENT TYPE: Journal

LANGUAGE: English

L20 ANSWER 103 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Radical polymerization of compds. containing C-C double bonds, photochem. or by electron beam, uses a (stabilized) photocatalyst and/or a sensitized photocatalyst including 21 organometallic compound in which the central coordinated atom is a transition element containing 1, 2, or 3 d electrons in (n-1) d orbitals (n = prin. quantum number) and in which ligands are coordinated to the central atom by 21 of C, N, P, As, Sb, O, S, Se, Te, B and/or simple unineg. ions of H, F, Cl, Br, I. The catalyst is used at 0.01% (based unsatd. monomer). The photosensitizer belongs to Ph ketones such as substituted benzophenones and the central coordinated atom belongs to 21 of Mn, Fe, Co, Ni, Cu, Mo, W, V, Pb, Zn, Zr, Pd, Ag, Au, Hg, Pt, Cr, Sn, U, Ge, and Ce. The composition may contain peroxy compds. Thus, to a 1-2 mm composite of 67 g styrenated polyester and 33 g glass fibers between Mylar films is added (at 1:1 to 10:1 molar ratio) 1% of peroxobis[N,N'-ethylenebis(salicylideneiminato)dimethylformamide cobalt(III)] and 1,2-diphenyl-2,2-dimethoxyethanone with UV irrads (100 W/cm) at 4 m/min for 15 s. Pultrusion may also be carried out.

ACCESSION NUMBER: 1991:472439 CAPLUS
 DOCUMENT NUMBER: 115:72439
 TITLE: Sensitization and stabilization of organometallic photocatalysts and photosensitizers in photochemical radical polymerization
 INVENTOR(S): Bellobono, Ignazio Renato
 PATENT ASSIGNEE(S): Chimia Prodotti e Processi S.r.l., Italy
 SOURCE: Eur. Pat. Appl., 11 pp.
 CODEN: EPOXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 421512	A1	19910410	EP 1990-202504	19900921
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE				
PRIORITY APPLN. INFO.: IT 1989-21914 A 19891003				
OTHER SOURCE(S): MARPAT 115:72439				

L20 ANSWER 105 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Kinetics and mechanisms of the following reactions in organic solvents have been studied: (i) formation of Schiff bases from aniline and derivs. of salicylaldehyde coordinated to Cu(II) and Zn(II); (ii) substitution of one β -diketonate ligand coordinated to Fe(III) by another β -diketonate ligand; (iii) formation of coordinated hemiacetals from β -diketonate ligands and alcs. in the coordination sphere of Fe(III); and (iv) formation of urethanes in the coordination sphere of Fe(III)(acac)2OR, where acac and OR are acetylacetonate and alkoxide anions. Such compds. as aniline, alcs., and phenylacetylene serve as catalysts of these reactions. Their catalytic action is explained by concerted processes of H-atom transfer in cyclic intermediate structures.

ACCESSION NUMBER: 1990:630706 CAPLUS
 DOCUMENT NUMBER: 113:230706
 TITLE: Concerted mechanisms of acid-base catalysis in the coordination sphere of metal complexes
 AUTHOR(S): Zamaraev, K. I.; Nekipelov, V. M.; Talsi, E. P.
 CORPORATE SOURCE: Inst. Catal., Novosibirsk, 630090, USSR
 SOURCE: Catalysis Letters (1990), 5(2), 127-41
 CODEN: CALEER; ISSN: 1011-372X
 DOCUMENT TYPE: Journal
 LANGUAGE: English

L20 ANSWER 104 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Trialkylamines are prepared in high yields with low ants. of heavy byproducts by reaction of an alc. or aldehyde with a mono- or dialkylamine at 150-275° in the presence of H and a recyclable catalyst initially consisting essentially of (1) a Cu compound selected from CuCO₃, CuO, or Cu(OH)₂; (2) a Zn compound selected from ZnCO₃, ZnO, or Zn(OH)₂; and (3) a metal compound selected from carbonates, oxides, and hydroxides of Group IIA metals such that at least a portion of the Cu or Zn compound is initially in the form of a carbonate. Thus, a mixture 1-octadecanol 100, CuCO₃ 1.55, Zn subcarbonate (5ZnO.2CO₂.4H₂O) 1.55, and BaCO₃ 0.2 g was flushed with N and then sparged with H at 0.1 ft³/h. The stirred mixture was heated to 210° and thereto a mixed gas of 0.1 ft³/h H and 0.4 ft³/h NMe₂ was sparged over 2 h to give octadecyldiethylamine 95.3, amide 0.1, and heavy products 0.2%. No example was given for amination of aldehydes with amines.

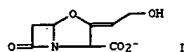
ACCESSION NUMBER: 1991:408082 CAPLUS
 DOCUMENT NUMBER: 115:8082
 TITLE: Catalysts and process for preparation of trialkylamines from a mono- or dialkylamine and an alcohol or aldehyde
 INVENTOR(S): Fong, Pak Y.; Smith, Kim R.; Sauer, Joe D.
 PATENT ASSIGNEE(S): Ethyl Corp., USA
 SOURCE: U.S., 6 pp. Cont.-in-part of U.S. Ser. No. 133,733.
 CODEN: USOXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 2
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4994620	A	19910219	US 1989-195883	19880519
US 4994622	A	19910219	US 1987-133733	19871216
PRIORITY APPLN. INFO.: US 1987-22095 B2 19870305				
US 1987-79522 B2 19870730				
US 1987-133733 A2 19871216				
US 1987-22047 B2 19870305				

L20 ANSWER 106 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Primary, secondary, and tertiary C2-4 amines are prepared by the reaction of C2-4 alcs. and/or C2-4 carbonyl compds. with NH₃ or amine, in a reducing atmosphere, preferably H, at 5-60 bar and 150-240°, in the presence of catalyst. The catalyst comprises 45-55% Ni, 45-55% Al and 0.05-5% Cu, Zn, Co, Mo and/or Si. The catalyst is leached with an alkali prior to use, and >2-20% of the Al and 0.5-10% of the Ni is present as oxide. The mol. ratio of H/NH₃ is 0.4:4 and that of NH₃/alkyl reactant 0.8:8. A catalyst consisting of an alloy containing Ni 46, Al 46, Cu 7 and Si 1% by weight was leached with 3% aqueous NaOH solution, at 50-60°, to remove 50% of the Al. Subsequently, the catalyst was treated with 2% MgCl₂ solution, as a promoter, washed with water, dried in a current of synthesis gas, and slowly heated to 225°. The catalyst was used in a fixed bed reactor, fed 1 mol acetone, 3.7 mmol NH₃ and 2.2 mmol H, at 221° and 8 bar. The rate of acetone feeding was 0.8 kg/h. The product consisted of 54.8% monoamine and 34.1% diamine (not specified). The conversion was 99.2%. Iso-PrOH (4.4%) was a byproduct.

ACCESSION NUMBER: 1990:497032 CAPLUS
 DOCUMENT NUMBER: 113:97032
 TITLE: Alkylamines by reductive amination of alcohols and oxo compounds
 INVENTOR(S): Kisgergely, Lajos; Patonai, Jozsef; Bozsay Hatas, Zsuzsanna; Antal, Jozsef; Budai, Laszlo; Voros, Istvan
 PATENT ASSIGNEE(S): Magyar Tudomanyos Akademia, Kozponti Kemiai Kutato Intezete, Hung.; Peti Nitrogenmuvek
 SOURCE: Hung. Teljes, 25 pp.
 CODEN: HUXXEU
 DOCUMENT TYPE: Patent
 LANGUAGE: Hungarian
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
HU 49111	A2	19890828	HU 1987-4466	19871005
HU 206667	B	19921228		
PRIORITY APPLN. INFO.: HU 1987-4466 19871005				



AB The Zn(II)-Tris system has a large catalytic effect on the hydrolysis and aminolysis of the clavulanate ion 1. In order to ascertain the mechanism of this catalysis the effects of other metal ions [Cd(II), Co(II), Cu(II), Ni(II), and Mn(II)], of amines structurally related to Tris, and of blocking the carboxylate group of 1 were investigated. From these studies it is concluded that only the Cd(II)-Tris and Co(II)-Tris systems have any substantial catalytic effect, although this is not as important as that of Zn(II)-Tris. Studies with Me clavulanate indicate that coordination of the metal ion by the carboxylate group is necessary. It is suggested that catalysis takes place via a ternary complex in which the metal ion plays a double role by placing the clavulanate ion and the amino alc. in the right position for the reaction and by lowering the pKa of the hydroxide group of Tris, which is coordinated with the metal ion, generating a strong nucleophile.

ACCESSION NUMBER: 1990:440237 CAPLUS
DOCUMENT NUMBER: 113:40237
TITLE: Studies on clavulanate. Part 3. Catalysis of hydrolysis and aminolysis of clavulanate acid by metal chelates
AUTHOR(S): Martin, Javier; Mendez, Rosa; Salto, Francisco; Castillo, Manuel
CORPORATE SOURCE: Dep. Bioquim. Biol. Mol., Univ. Leon, Leon, 24071, Spain
SOURCE: Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1990), (1), 43-6
CODEN: JCPKHH; ISSN: 0300-9580
DOCUMENT TYPE: Journal
LANGUAGE: English

AB Primary and secondary alcs. and diols are condensed with amines at elevated temperature over a nonacidic cationic hydroxyapatite catalyst comprising (M₂)_x(PO₄)_y(OH)_z (M = Ca, Mg, Sr, La, Ce, Fe, Cu, Zn, Al, B, Ga; x/y is .apprx.1.3 to .apprx.2.4 when a = 2; x/y is .apprx.0.87 to .apprx.1.6 when a = 3; z/y is .apprx.1 to .apprx.3). Thus, EtOH and piperidine (I) were passed over a calcium hydroxyapatite catalyst (Ca/P = 1.7) (preparation given) maintained at 357° at GHSV = 160 h⁻¹ to give 84% conversion of I with 98% selectivity for N-ethylpiperidine.

ACCESSION NUMBER: 1990:235192 CAPLUS
DOCUMENT NUMBER: 112:235192
TITLE: Catalytic animation of alcohols and diols using non-acidic hydroxyapatite catalysts
INVENTOR(S): Farris, Gene E.; Pierantozzi, Ronald
PATENT ASSIGNEE(S): Air Products and Chemicals, Inc., USA
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4900832	A	19900213	US 1988-227569	19880802

PRIORITY APPLN. INFO.: US 1988-227569 19880802
OTHER SOURCE(S): CASREACT 112:235192

AB Aromatic compds. are alkylated by C₂₆ alkylating agents, i.e. alcs., alkyl halides, or olefins, in the presence of a layered catalyst comprising a layered metal oxide (e.g. TiO₂) and pillars of an oxide (e.g. SiO₂) separating the layers of the metal oxide. Thus, C₂₆O₃

and TiO₂ were fired at 650° 3 times, with intermediate grindings between firings and the fired material was ball-milled, ion-exchanged with 1N NH₄NO₃ at reflux and filtered. The NH₄⁺-exchanged material was refluxed in neat octylamine with removal of H₂O, filtered, washed with EtOH, air-dried, treated at 80° with Si(OEt)₄, filtered, dried, treated with H₂O and then calcined at 500° in flowing air to give a layered material catalyst containing 23 ppm Cs, 29.0 weight% Ti and 44.9 weight% SiO₂. Benzene was alkylated by *n*-C₁₄ olefin (Shell Neodene-14) for 6 h at 400°F and 400 psig N in the presence of the catalyst prepared above to give 60 weight% lube range material vs. 14.0 and 11.0 weight% when conventional catalysts were used.

ACCESSION NUMBER: 1990:440122 CAPLUS
DOCUMENT NUMBER: 113:40122
TITLE: Preparation of long chain alkyl aromatic compounds by alkylation of aromatic compounds
INVENTOR(S): Aufdenbrink, Brent A.; Kresge, Charles T.; Le, Quang N.; Shin, Joonup; Wong, Stephen S.
PATENT ASSIGNEE(S): Mobil Oil Corp., USA
SOURCE: U.S., 9 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4912277	A	19900327	US 1989-358105	19890530
US 5043508	A	19910827	US 1990-483690	19900223
CA 2017078	AA	19901130	CA 1990-2017078	19900517
EP 400857	A1	19901205	EP 1990-305513	19900521
EP 400857	B1	19930922		
RU 9055747	B1	19901206	AU 1990-55747	19900521
AU 623257	B2	19920507		
JP 03034939	A2	19910214	JP 1990-141217	19900530
JP 2744511	B2	19980428		

PRIORITY APPLN. INFO.: US 1989-358105 A2 19890530
US 1990-483690 A 19900223

AB Under the Federal Comprehensive Environmental Response, Compensation, and Liability Act, the EPA is promulgating final reportable quantities (RQ) for 258 hazardous substances and hazardous waste streams. NH₄ thiosulfate is removed from the list of hazardous substances since the median lethal concentration is well above 500 mg/L for aquatic toxicity. Also included in this

final rule is replacement of the registered trademark Gelthane with the generic name difocal, as several companies manufacture this substance.

ACCESSION NUMBER: 1990:83178 CAPLUS
DOCUMENT NUMBER: 112:83178
TITLE: Reportable quantity adjustments; delisting of ammonium thiosulfate
CORPORATE SOURCE: United States Environmental Protection Agency, Washington, DC, 20460, USA
SOURCE: Federal Register (1989), 54(155), 33426-84, 14 Aug 1989
CODEN: PEREAC; ISSN: 0097-6326
DOCUMENT TYPE: Journal
LANGUAGE: English

L20 ANSWER 111 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The title catalyst (I), prepared by treating CuSO₄ with Zn in H₂O, gave high yields (generally 86-95%) of aromatic amines in the title reduction. Thus, 2-, 3- and 4-MeC₆H₄NO₂ were treated with H₂NNH₂ in EtOH in the presence of I to give 90, 90 and 92% 2-, 3- and 4-MeC₆H₄NH₂ resp.
 ACCESSION NUMBER: 1990:55189 CAPLUS
 DOCUMENT NUMBER: 112:55189
 TITLE: Reduction of aromatic nitro compounds with hydrazine catalyzed by activated zinc-copper
 AUTHOR(S): Han, Byung Hee; Shin, Dae Hyun; Lee, Hyun Ro; Ro, Bong Ho
 CORPORATE SOURCE: Coll. Nat. Sci., Chungnam Natl. Univ., Daejeon, 302-764, S. Korea
 SOURCE: Bulletin of the Korean Chemical Society (1989), 10(3), 315-16
 CODEN: BKCSDE; ISSN: 0253-2964
 DOCUMENT TYPE: Journal
 LANGUAGE: English

L20 ANSWER 112 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Methylation of iso-PrNH₂ with H₂/CO over Cu/ZnO catalysts was carried out at moderate temps. and pressures, with higher temps. favoring higher yields and lower temps. giving higher selectivity of monomethylation. The catalyst was used to hydrogenate Et₂NHCO, and to react iso-PrOH with iso-PrNH₂ and NH₃ with H₂/CO. Catalyst doping with Fe and the use of a Pd/SiO₂ catalyst were also studied.
 ACCESSION NUMBER: 1990:38571 CAPLUS
 DOCUMENT NUMBER: 112:38571
 TITLE: Synthesis of aliphatic amines over methanol synthesis catalysts
 AUTHOR(S): Vedage, Gamin A.; Herman, Richard G.; Klier, Kamil
 CORPORATE SOURCE: Air Prod. and Chem., Inc., Allentown, PA, USA
 SOURCE: Chemical Industries (Dekker) (1988), 33(Catal. Org. React.), 149-76
 CODEN: CHEIDI; ISSN: 0737-8025
 DOCUMENT TYPE: Journal
 LANGUAGE: English

L20 ANSWER 113 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Amines are prepared by catalytic amination of alcs. or aldehydes with NH₃ or primary or secondary amines over a catalyst composition of (a): Cu/(Cr, Mn, Fe, Zn)/Pt Group; (b): Cu/Co/Pt Group; (c): Cu/(Cr, Mn, Fe, Co, Ni Zn)/Pt Group/alkali and alkaline earth metal; or (d): Cu/(Cr, Mn, Fe, Co, Ni, Zn)/Pt Group/(Al, W, Mo). A flask containing lauryl alc. and 0.25 weight% ternary catalyst composition of 4:1:0.05 Cu-Cr-Pd (preparation given) was purged with N₂, H₂ introduced at 100°, followed by Me₂NH and H₂ at 200° for 5 h to give a mixture containing lauryldimethylamine 95.6, unreacted alc. 1.5, and others 2.9 weight%, vs. 89.0, 5.3, and 5.7 weight%, resp., with a 4:1 Cu-Ni catalyst composition
 ACCESSION NUMBER: 1989:594101 CAPLUS
 DOCUMENT NUMBER: 111:194101
 TITLE: Process for preparing substituted amines
 INVENTOR(S): Abe, Hiroshi; Aikawa, Jun; Okabe, Kazuhiko; Sotoya, Kohshiro
 PATENT ASSIGNEE(S): Kao Corp., Japan
 SOURCE: Eur. Pat. Appl., 46 pp.
 CODEN: EPXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 312253	A2	19890419	EP 1988-309343	19881007
EP 312253	A3	19900411		
EP 312253	B1	19930512		
R: DE, ES				
JP 01102045	A2	19890419	JP 1987-261367	19871016
JP 03004536	B4	19910123		
JP 02000232	A2	19900105	JP 1988-251674	19881005
JP 04070301	B4	19921110		
JP 02000233	A2	19900105	JP 1988-251675	19881005
JP 04070302	B4	19921110		
JP 02000234	A2	19900105	JP 1988-251676	19881005
JP 04070303	B4	19921110		
EP 485371	A1	19920513	EP 1992-200338	19881007
EP 485371	B1	19950614		
R: DE, ES				
EP 487514	A1	19920527	EP 1992-200340	19881007
EP 487514	B1	19950614		
R: DE, ES				
EP 489722	A1	19920610	EP 1992-200339	19881007
EP 489722	B1	19950201		
R: DE, ES				
ES 2054819	T3	19940816	ES 1988-309343	19881007
ES 2067290	T3	19950316	ES 1992-200339	19881007
ES 2073235	T3	19950801	ES 1992-200338	19881007
ES 2073236	T3	19950801	ES 1992-200340	19881007
US 5266730	A	19931130	US 1990-529928	19900529
PRIORITY APPLN. INFO.:			JP 1987-261366	A 19871016
			JP 1987-261367	A 19871016
			JP 1987-261368	A 19871016
			JP 1987-261369	A 19871016
			US 1988-255910	B1 19881007

L20 ANSWER 113 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

L20 ANSWER 114 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Reduction catalysts are prepared by precipitating Ni, Co, or Pd on Zn, Fe, or Al. This is carried out by contacting the latter metals with solns. of compds. of Ni, Co, or Pd. The solvents are preferably selected from those which will also dissolve the Zn, Fe, or Al compds. formed in the reaction. The catalysts are more effective than Zn, Fe, or Al by themselves. A mixture of 20 g Al granules and 17 g NiCl₂·6H₂O in 100 mL EtOH was heated 5 min at 60-65°, followed by cooling to 20°, addition of 54 g PhCH₂Cl, 200 mL H₂O, and 300 mL 15% H₂SO₄, and stirring 3 h to give 36.6 g PhMe (94.1% yield). In the presence of NiCl₂·6H₂O alone the yield was 4.8%.

ACCESSION NUMBER: 1989:553332 CAPLUS
 DOCUMENT NUMBER: 111:153332
 TITLE: Metal-coated metallic particles as reduction catalysts
 INVENTOR(S): Mailat, Tamas; Petro, Jozsef; Bodnar, Zsolt
 PATENT ASSIGNEE(S): Magyar Tudomanyos Akademia, Kutatas- es Szervezetelemzo Intezet, Hung.
 SOURCE: Hung. Teljes, 13 pp.
 CODEN: HUXKBU
 DOCUMENT TYPE: Patent
 LANGUAGE: Hungarian
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
HU 46636	A2	19881128	HU 1986-3920	19860912
HU 202463	B	19910328		

PRIORITY APPLN. INFO.: HU 1986-3920 19860912

L20 ANSWER 116 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Trialkylamines are prepared by reacting a lower mono- or dialkylamine with a C8-22 primary alc. in the presence of H at 180-300° in the presence of a Cu-Zn-alkaline earth metal base-containing catalyst while removing water formed in the reaction. 1-Dodecanol 100, CuO powder 1.0, ZnO powder 1.0, and Ba(OH)₂·8H₂O 0.2 g were heated to 100° under N, the mixture was sparged with H and heated to 200°, and Me₂NH (115 g) was fed into the reaction mixture over 4 h, producing dodecyltrimethylamine 93.5, dialkylmethylamine 3.2, and dodecanol 1.4 area percent.

ACCESSION NUMBER: 1989:40803 CAPLUS
 DOCUMENT NUMBER: 110:40803
 TITLE: Catalysts and process for preparation of trialkylamines from C8-22 primary alcohols and lower dialkylamines
 INVENTOR(S): Fong, Pak Yiu; Smith, Kim Renae; Sauer, Joe Dean
 PATENT ASSIGNEE(S): Ethyl Corp., USA
 SOURCE: Eur. Pat. Appl., 14 pp.
 CODEN: EPXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 2
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 281417	A2	19880907	EP 1988-301920	19880304
EP 281417	A3	19891227		

R: AT, BE, CH, DE, ES, FR, GB, IT, LI, LU, NL, SE
 US 4994622 A 19910219 US 1987-133733 19871216
 JP 01013060 A 19890117 JP 1988-51454 19880304
 US 4957893 A 19900918 US 1989-365213 19890612
 US 1987-22095 A 19870305
 US 1987-79522 A 19870730
 US 1987-133733 A 19871216
 US 1987-22047 19870305

PRIORITY APPLN. INFO.:
 OTHER SOURCE(S): CASREACT 110:40803

L20 ANSWER 115 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Amines are prepared by contacting alcs. and/or ketones and/or aldehydes with NH₃ and H in the presence of a catalyst comprising Co 6-47, Cu 75-95, and a third component (selected from Fe and/or Sn and/or Zr) 1-16% (calculated in mol% on an oxide-free basis). These catalysts offer equal or higher reductive amination activity at a lower cost than do conventional high Co content catalysts. A catalyst was prepared containing Co 15.0, Cu 79.0, and Zr 6.00 mol% and having surface area 30.0 m²/g and total H adsorption 1.055 cm³/g after reduction in 18% H in N at 175° for 4 h. The catalyst was contacted with ethanolamine, NH₃, and H (750 psi) (NH₃/ethanolamine mol ratio 8.5) at 170°/1500 psi with residence time 10 min, giving ethylenediamine 47, diethylenetriamine 17, triethylenetetramine 13, piperazine 12, aminoethylpiperazine 2, and aminoethylethanolamine 11%.

ACCESSION NUMBER: 1989:195148 CAPLUS
 DOCUMENT NUMBER: 110:195148
 TITLE: Process and low-cost amination catalysts for the manufacture of amines from alcohols, aldehydes or ketones
 INVENTOR(S): Bowman, Robert G.
 PATENT ASSIGNEE(S): Dow Chemical Co., USA
 SOURCE: U.S., 5 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4806690	A	19890221	US 1985-732978	19850513
US 4891349	A	19900102	US 1988-264908	19881107
WO 9009368	A1	19900823	WO 1989-US694	19890217

W: BR, JP, KR
 BR 8907390 A 19910514 BR 1989-7390 19890217
 JP 03503977 T2 19910905 JP 1989-502787 19890217
 EP 384047 A1 19900829 EP 1989-301602 19890220
 R: BE, DE, ES, FR, GB, IT, NL, SE
 CN 1045039 A 19900905 CN 1989-101846 19890221
 US 1985-732978 A3 19850513
 WO 1989-US694 A 19890217

PRIORITY APPLN. INFO.:
 OTHER SOURCE(S): CASREACT 110:195148

L20 ANSWER 117 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Now in waste gases containing As compds. is removed with a reducing agent, e.g., NH₃, in the presence of a reduction catalyst comprising Ti, Zr selected from the group of As and Mn, and Zr base metal selected from the group of V, W, Mo, Cu, Fe, Cr, Co, Ni, Zn, and Sn. The catalyst contains As approx. 0.01-15 weight parts based on As₂O₃, or Mn 1-85 weight parts based on MnO₂, or both, Zr base metal 0.05-80 weight parts based on V₂O₅, WO₃, MoO₃, CuO, Fe₂O₃, Cr₂O₃, Cr₃O₄, NiO, ZrO, or SnO₂ in relation to Ti 100 weight parts based on TiO₂. This catalyst resists catalyst poisoning by As compds. in waste gases and is durable over long time periods. Thus, metatitanic acid as an intermediate from TiO₂ production by H₂SO₄ process was neutralized, filtered, and washed with water to give metatitanic acid cake. A 800-kg portion of metatitanic acid (based on TiO₂) was added to 8 kg 67.5% HNO₃ solution. The resulting solution was spray dried, calcined at 450° for 3 h, and pulverized to give TiO₂ powder (average size 2μ). An aqueous solution containing monoethanolamine 300 L, NH₄ tungstate 100, and arsenious acid 2.1 kg were mixed with TiO₂ powder 800, poly(vinyl alc.) 50, 5-mm long and 9-μ diameter glass fibers 100, and water 100 L and the mixture was kneaded, pressed through a vacuum screw extruder to give a honeycomb mold, which was dried in air at 100° for 5 h, cut at both axial ends and calcined at 450° for 3 h to give a honeycomb (cell pitch 7.4 mm, wall thickness 1.35 mm). Water was added to 19.2 kg oxalic acid and 7.7 kg NH₄VO₃ to give 40 L aqueous solution containing 150-g V₂O₅/L, which was diluted with water to 17.9-g V₂O₅/L. The honeycomb was immersed in the solution to give a coated honeycomb which was dried in air at room temperature for 2.5 h, dried at 100° for 5 h to give a honeycomb catalyst containing Ti, W, V, and As at 89.3, 9.9, 0.6, and 0.23% based on TiO₂, WO₃, V₂O₅, and As₂O₃, resp., based on the total weight of metal components in the catalyst. The prepared catalyst was cut into a honeycomb of 30 mm in length and having 9 openings extending into 3 × 3 cells. A mixture of N₂ 200, NH₃ 200, SO₂ 800, As₂O₃ 25 ppm, 0.10, water vapor 10, and CO₂ 12 volume% with balance of N was contacted with the honeycomb catalyst at 380° and at 4700 h- space velocity for 5 h. The resulting denitration rate was 93.5%.

ACCESSION NUMBER: 1988:209453 CAPLUS
 DOCUMENT NUMBER: 108:209453
 TITLE: Catalyst and method for removing nitrogen oxides from waste gases
 INVENTOR(S): Rikimaru, Hiroaki; Nakatsuji, Tadao; Umaba, Toshikatsu; Nagano, Kazuhiko; Mishina, Kazuya; Shimizu, Hiromitsu; Nojima, Shigeru; Iida, Kozo; Ohayashi, Yoshiaki; et al.
 PATENT ASSIGNEE(S): Sakai Chemical Industry Co., Ltd., Sakai, Japan; Mitsubishi Heavy Industries, Ltd.; Mitsubishi Petrochemical Co., Ltd.; Mitsubishi Petrochemical Engineering Co.; Catalysts and Chemicals Industries Co., Ltd.
 SOURCE: Eur. Pat. Appl., 20 pp.
 CODEN: EPXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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L20 ANSWER 117 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

EP 257307 A1 19880302 EP 1987-110657 19870723

EP 257307 B1 19920429

R: AT, BE, DE, FR, GB, IT, NL, SE

JP 63031526 A2 19880210 JP 1986-173813 19860725

JP 63036838 A2 19880217 JP 1986-178725 19860731

JP 63072341 A2 19880402 JP 1986-216356 19860913

JP 63072343 A2 19880402 JP 1986-216357 19860913

AT 75417 E 19920515 AT 1987-110657 19870723

CA 1307251 A1 19920908 CA 1987-542955 19870724

US 4952381 A 19900828 US 1988-284764 19881212

PRIORITY APPLN. INFO.:

JP 1986-173813 A 19860725

JP 1986-178725 A 19860731

JP 1986-216356 A 19860913

JP 1986-216357 A 19860913

EP 1987-110657 A 19870723

US 1987-77868 B3 19870723

L20 ANSWER 118 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A thermosetting PhOH-furan resin binder is used to coat foundry sand for substantial eliminating the use of hexamethylenetetraamine. Thus, a resin was prepared, in a reflux condenser, from a mixture containing PhOH

1200, aqueous 50% HCHO 1200, and aqueous 25% Zn acetate 60 g by heating 4 h at 97-100° until free HCHO was 6.45%, dehydrating in vacuum at .apprx.50° to remove 549 g volatiles, and then reacting with 800 g furfuryl alc. for .apprx.3.3 h at 90-100°. Hot SiO2 sand 1000, the resin 44, Araxax C lubricant 1.5, aqueous 50% NH4NO3 0.4 g, and water 45 mL were mixed, and the mixture was hot-pressed into tensile specimens, cooled, and tested. The tensile strength was 318 psi and ignition loss 3.04%, compared with 420 psi and 3.36% for a conventional shell using a novolak resin.

ACCESSION NUMBER: 1988:136200 CAPLUS

DOCUMENT NUMBER: 108:136200

TITLE: Foundry shell core and mold composition

INVENTOR(S): Armbruster, David R.; Johnson, Calvin K.

PATENT ASSIGNEE(S): Acme Resin Corp., USA

SOURCE: U.S., 6 pp.

CODEN: USXKAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4713294	A	19871215	US 1986-866438	19860523
CA 1297740	A1	19920324	CA 1987-537503	19870520
PRIORITY APPLN. INFO.:			US 1986-866438	A 19860523

L20 ANSWER 119 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB N-Alkylamines are prepared by reaction of aromatic, alicyclic or heterocyclic amines with aliphatic alcs. (as alkylating agents) in the presence of Cu-Zn/Al2O3 catalyst in a fixed-bed reactor at 150-300°/1-10 kg/cm2. The catalyst contains 10-50% Cu and 10-70% Zn. Thus, a 1:2.24 (mol. ratio) PhNH2-MeOH mixture was passed over the catalyst at 250°, 1 kg/cm2, and 0.4 h-1 liquid space velocity to give 94.9% yield N-methylaniline at 99% once-through PhNH2 conversion.

ACCESSION NUMBER: 1988:133821 CAPLUS

DOCUMENT NUMBER: 108:133821

TITLE: Preparation of N-alkylamines

INVENTOR(S): Xu, Bingsheng; Ding, Li; Sun, Yan; Lou, Zhenhua; Wang, Weimin

PATENT ASSIGNEE(S): Fushun Petrochemical Research Institute, Sinopec, Peop. Rep. China

SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 6 pp.

CODEN: CNXKEV

DOCUMENT TYPE: Patent

LANGUAGE: Chinese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CN 85100326	A	19860917	CN 1985-100326	19850401
CN 85100326	B	19871202		
PRIORITY APPLN. INFO.:			CN 1985-100326	19850401

L20 ANSWER 120 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A procedure for preparing a secondary amine was characterized in that an alc. or an aldehyde is reacted with a primary amine in the presence of a catalyst of Cu, Ni and a metallic element of Pt group VIII, at atmospheric pressure to 6 bar (5 kg/cm2 gage) at 150-250° with removal of reaction formed H2O. A mixture of C20 Guerbet alc., laurylamine, and 4:1:0.4 Cu-Ni-Ru catalyst was heated up to 190° 4.5 h to give a product containing 98.1% secondary amine. If Ru is omitted, the product contained 29.8% secondary amine and if Ni is omitted, 25.6%.

ACCESSION NUMBER: 1987:597532 CAPLUS

DOCUMENT NUMBER: 107:197532

TITLE: Preparation of secondary amines from alcohols or aldehydes and primary amines

INVENTOR(S): Yokota, Yukinaga; Sawamoto, Yuzi; Taniguchi, Hideaki; Okabe, Kazuhiko

PATENT ASSIGNEE(S): Kao Corp., Japan

SOURCE: Ger. Offen., 8 pp.

CODEN: GWXKEV

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3641666	A1	19870702	DE 1986-3641666	19861205
DE 3641666	C2	19990422		
JP 62149648	A2	19870703	JP 1985-296112	19851225
JP 03054931	B4	19910821		
US 4792622	A	19881220	US 1986-934230	19861121
ES 2001465	A6	19880516	ES 1986-3533	19861219
FR 2592041	A1	19870626	FR 1986-18178	19861224
FR 2592041	B1	19900615		
PRIORITY APPLN. INFO.:			JP 1985-296112	A 19851225

L20 ANSWER 121 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Cinnamic acid esters useful as starting materials for perfumes, agrochems., and photosensitive polymers are prepared from styrene, CO, alcs., and O in the presence of catalysts containing Pd compds. and Cu compds., and the catalysts are separated as solids from the reaction mixts., oxidized in the presence of inorg. anions, and used repeatedly. Me cinnamate was prepared using Pd chloride and CuCl₂ as the catalyst, which was recovered for Pd 78, Cu 92, and Cl 981.

ACCESSION NUMBER: 1987:578618 CAPLUS
 DOCUMENT NUMBER: 107:178618
 TITLE: Recycling of catalysts
 INVENTOR(S): Yamamoto, Yoshihiro; Aoki, Shinobu; Takagi, Usaji
 PATENT ASSIGNEE(S): Mitsui Toatsu Chemicals, Inc., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.
 CODEN: JKOXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 62081350	A2	19870414	JP 1985-219034	19851003
PRIORITY APPLN. INFO.:			JP 1985-219034	19851003

L20 ANSWER 122 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB RnCGHS-nNHR1 (I, R = H, halo, alkyl, alkoxy; R1 = alkyl, Ph; n = 1-3) were prepared by treating I (R1 = H) with R1OH over catalysts containing MgO and 22 metal oxides chosen from Zn, Cd, Al, Fe, Ca, Ni, Cu, and Ti. A mixture of 1:1 PhNH₂ and MeOH was passed at 6.5 g/h through 1 g catalyst of 90:5:5 MgO-ZnO-Al₂O₃ at 320° for 5 h to give only PhNHMe with 94.3% conversion.

ACCESSION NUMBER: 1987:439359 CAPLUS
 DOCUMENT NUMBER: 107:39359
 TITLE: Aromatic secondary amino compounds
 INVENTOR(S): Okuno, Hiroshi; Yamamoto, Moriharu
 PATENT ASSIGNEE(S): Nihon Nohyaku Co., Ltd., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
 CODEN: JKOXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 61291551	A2	19861222	JP 1985-134689	19850620
JP 06002716	B4	19940112		
PRIORITY APPLN. INFO.:			JP 1985-134689	19850620

L20 ANSWER 123 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Unsatd. fatty acids are hydrogenated to unsatd. fatty alcs. in the presence of an excess of a trialkylamine, using a Zn -Cr catalyst (optionally activated with Ni and/or Cu) at >570 K and H pressure >18 MPa. The neutralization with the trialkylamine prevents the loss of catalytic activity when free fatty acids are hydrogenated. Thus, 200 g olein [37220-82-9]/h and 20 g Me₃N/h were added to a hydrogenation reactor which contained a catalyst comprising ZnO and Cr₂O₃ and was heated at 585-595°. The reactor was operated satisfactorily for 14 days. When the process was repeated without Me₃N, the catalyst became inactive after 3 days.

ACCESSION NUMBER: 1985:205748 CAPLUS
 DOCUMENT NUMBER: 102:205748
 TITLE: Unsaturated fatty alcohols
 INVENTOR(S): Aring, Heinz; Busch, Kurt; Franke, Peter; Konetzke, Gerhard; Tietz, Wolfgang; Weidemann, Reinhold
 PATENT ASSIGNEE(S): VEB Deutsches Hydrierwerk, Ger. Dem. Rep.
 SOURCE: Ger. (East), 8 pp.
 CODEN: GEKXAS
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DD 213429	A1	19840912	DD 1983-247119	19830110
PRIORITY APPLN. INFO.:			DD 1983-247119	19830110

L20 ANSWER 124 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB ZnO-Cu-alkali metal and Cu- and Pd-metal oxide catalysts were used in the alkylation of NH₃ by H-CO and of alkylamines by alcs. and H-CO. Thus, NH₃ was treated with H-CO over ZnO-Cu-Na to give MeNH₂. The N-alkylation of Me₂CHNH₂ by EtOH was catalyzed by Pd/SiO₂ and gave EtNHCHMe₂.

ACCESSION NUMBER: 1985:95238 CAPLUS
 DOCUMENT NUMBER: 102:95238
 TITLE: Catalyst and method for production of alkylamines
 INVENTOR(S): Klier, Kamil; Herman, Richard G.; Vedage, Ganini A.
 PATENT ASSIGNEE(S): Lehigh University, USA
 SOURCE: Eur. Pat. Appl., 53 pp.
 CODEN: EPXKDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 2
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 127874	A2	19841212	EP 1984-106136	19840529
EP 127874	A3	19851002		
R: BE, DE, FR, GB, NL				
US 4480131	A	19841030	US 1983-500037	19830601
US 4642381	A	19870210	US 1983-555579	19831128
US 5021233	A	19910604	US 1989-371425	19890626
PRIORITY APPLN. INFO.:			US 1983-500037	A 19830601
			US 1983-555579	A 19831128
			US 1983-500073	A2 19830601
			US 1983-558366	B1 19831205
			US 1985-759494	B1 19850726
			US 1986-919592	B1 19861017
			US 1987-70593	B1 19870707
			US 1987-135571	A3 19871227

L20 ANSWER 125 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Secondary and tertiary amines were prepared by alkylating primary amines using SCl_2 alics. with 0.5:1 to 20:1 molar ratios, at 1-100 atm and 50-250°, using either Cu-ZnO or Cu or Pd and at least 1 of Zn oxide, alumina, and Cr oxide as catalysts. Thus $MeNH_2$ was treated with $BuOH$ in a 2:1 molar ratio at 26 atm and 190°, to give 52.4% $BuOH$ conversion with 95.5% selectivity to $MeNH_2$.

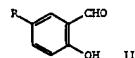
ACCESSION NUMBER: 1985:45475 CAPLUS
 DOCUMENT NUMBER: 102:45475
 TITLE: Selective production of di- and trialkylamines
 INVENTOR(S): Klier, Kamil; Herman, Richard G.; Vedage, Ganini A.
 PATENT ASSIGNEE(S): Lehigh University, USA
 SOURCE: U.S., 7 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 2
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4480131	A	19841030	US 1983-500037	19830601
US 4642381	A	19870210	US 1983-555579	19831128
EP 127874	A2	19841212	EP 1984-106136	19840529
EP 127874	A3	19851002		
R: BE, DE, FR, GB, NL				
JP 60041544	A2	19850305	JP 1984-109777	19840531
US 4843101	A	19890627	US 1987-135571	19871221
US 5021233	A	19910604	US 1989-371425	19890626
PRIORITY APPLN. INFO.:			US 1983-500037	A1 19830601
			US 1983-500073	A2 19830601
			US 1983-555579	A 19831128
			US 1983-558366	A1 19831205
			US 1985-759494	A1 19850726
			US 1986-919592	A1 19861017
			US 1987-70593	A1 19870707
			US 1987-135571	A3 19871227

L20 ANSWER 126 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The title compds. $HC.tpbond.CCH_2OSiR_2Me$ (I, R = Me_2CH , Me_2CHCH_2 , pentyl) were prepared in 25-30% yields by dehydrocondensation of $HC.tpbond.CCH_2OH$ with Me_2SiH in the presence of $ZnCl_2$. The aminomethylation, hydrosilylation and hydration of I were studied. Thus, aminomethylation of I with $HCHO$ and Et_3NH in the presence of $CuCl$ gave 35-80% $Me_2SiOCH_2C.tpbond.CCH_2NH_2$.

ACCESSION NUMBER: 1984:423574 CAPLUS
 DOCUMENT NUMBER: 101:23574
 TITLE: Synthesis of methyldi(iso)alkylpropargyloxysilanes with isopropyl, isobutyl, and pentyl substituents of silicon
 AUTHOR(S): Movsumzade, M. M.; Guseinov, Sh. O.; Guido Yanez, Q.; Karaev, S. F.
 CORPORATE SOURCE: AZINEFTEKHIM, Baku, USSR
 SOURCE: Doklady - Akademiya Nauk Azerbejdzhanskoi SSR (1983), 39(8), 41-5
 CODEN: DAZRA7; ISSN: 0002-3078
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian
 OTHER SOURCE(S): CASREACT 101:23574

L20 ANSWER 127 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 G1



AB The kinetics of Schiff base formation between aniline (I) and Cu, Na and Zn complexes of salicylaldehydes II (R = H, Cl, Br, NO₂) and uncomplexed II (R = H, NO₂) were examined. The reaction with the metal complexes proceeded via attack by uncoordinated I on the ligand to form the carbinolamine, which was then dehydrated. The attack occurred outside the plane of the chelate ring. The mechanism with II and their complexes were similar, the difference in rates resulting from different stabilities of the carbinolamine intermediates in the free and coordinated states. The mechanism of catalysis of alics and amines was described.

ACCESSION NUMBER: 1984:406528 CAPLUS
 DOCUMENT NUMBER: 101:6528
 TITLE: Spectroscopic study of the kinetics and mechanism of formation of Schiff bases in the coordination sphere of transition metals
 AUTHOR(S): Talzi, E. P.; Mekipelov, V. M.; Zamaraev, K. I.
 CORPORATE SOURCE: Inst. Katal., Novosibirsk, USSR
 SOURCE: Zhurnal Fizicheskoi Khimii (1984), 58(2), 273-91
 CODEN: ZFKH99; ISSN: 0044-4537
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 128 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB CrO_3 or H_2CrO_4 or its salts are treated with an alic., sugar, hydroxy acid, aldehyde, ketone, or amine and used with Cu and Zn compds. to prepare a catalyst for the preparation of $MeOH$ [67-56-1] from synthesis gas. Thus, H_2O containing 2 g CrO_3 and 1.84 g glycerol (I) [56-81-5] was refluxed 1 h, mixed with a precipitate from NH_4HCO_3 and Cu and Zn nitrates, dried, calcined at 370°, ground, and used to prepare a catalyst containing 56:42:2 Cu-Zn-Cr. The catalysts was heated at 140° in N and used at 260° for the preparation of $MeOH$ from H₂, CO₂, CO, CH₄ 3.5, and N 0.5%. The reaction was interrupted periodically to heat the catalyst at 360°. During initial $MeOH$ preparation and after each heating, the effluent contained 12.4-13.8% $MeOH$, compared with 9.3-9.7 for a catalyst prepared without I.

ACCESSION NUMBER: 1983:524417 CAPLUS
 DOCUMENT NUMBER: 99:124417
 TITLE: Methanol synthesis catalyst
 PATENT ASSIGNEE(S): Mitsubishi Gas Chemical Co., Inc., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
 CODEN: JKXKAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 58067348	A2	19830421	JP 1981-167718	19811020
JP 61007332	B4	19860305		
GB 2110557	A1	19830622	GB 1982-29786	19821019
GB 2110557	B2	19850109		
PRIORITY APPLN. INFO.:			JP 1981-167718	A 19811020

L20 ANSWER 129 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Urethanes were prepared by reaction of aromatic amine compds., CO, and organic hydroxy compds. in the presence of mol. O or aromatic nitro compds., Pt Group catalysts, and lamellar compds. (cocatalysts) between Cu, Zn, Ni, V, Cr, Mn, or Fe Group halides and graphite. Thus, a mixture of 50 mmol PhNH₂, 50 mL EtOH, 0.7 mg Pd black, 3.5 g of a lamellar compound of 15 weight % FeCl₃-graphite, 80 kg/cm² CO, and 5 kg/cm² was autoclaved 1 h at 160° to give 66% PhNHCO₂Et. Also, prepared were PhNHCO₂Me and 2,5-(EtO₂CNH)2C₆H₃Me.
 ACCESSION NUMBER: 1983:405375 CAPLUS
 DOCUMENT NUMBER: 99:5375
 TITLE: Urethane compounds
 PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
 CODEN: JXOXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 57200349	A2	19821208	JP 1981-84605	19810602
JP 63032346	B4	19880629		

PRIORITY APPLN. INFO.: JP 1981-84605 19810602

L20 ANSWER 130 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 G1 For diagram(s), see printed CA Issue.
 AB MeCH(OH)CH₂CH₂NMe₂ (I) or MeCH(OH)CH₂CH(NMe₂)Me are converted, in the gas phase [in a flow reactor over prehydrogenated CuO(Cr₂O₃)ZnO] to AcEt or AcPr, resp. The mechanism for I involves dehydrogenation to AcCH₂CH₂NMe₂ (II) which is deaminated to give AcCH:CH₂ (III) which is hydrogenated to AcEt by more I (to give II) in a Cu catalyzed H transfer reaction. II and III are observed chromatog. in the reaction mixture, II is deaminated under the reaction conditions by thermal and catalytic processes. IV (n = 3-5) and RCH(OH)CH₂CH(NMe₂)R₁ (R = Me, Et, Ph; R₁ = H, Me) also gave ketonic products under these conditions; amino alcs. with primary OH groups give resinous products and compds. containing functional groups on tert-C atoms lead to fragmentation.
 ACCESSION NUMBER: 1982:423031 CAPLUS
 DOCUMENT NUMBER: 97:23031
 TITLE: Transformation of 1,3-amino alcohols to ketones on copper
 AUTHOR(S): Bartok, Mihaly; Sirokman, Geza; Molnar, Arpad
 CORPORATE SOURCE: Dep. Org. Chem., Jozsef Attila Univ., Szeged, Hung.
 SOURCE: Journal of Molecular Catalysis (1982), 14(3), 379-82
 CODEN: JMCADS; ISSN: 0304-5102
 DOCUMENT TYPE: Journal
 LANGUAGE: English

L20 ANSWER 131 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB A number of compds., mostly metal salts and amines, catalyzed the known reaction of alcs., amines, and urea to give urethanes. Thus, 1163 g 2-ethylhexylamine, 541 g urea, 1153 g MeOH, and 5.0 g Zn octanoate were heated 4.5 h at 180° with distillation of NH₃ to give 89% BuCH₂CH₂CH₂NHCO₂Me. Among 21 other catalysts used (for preparation of 20 other urethanes) were TiCl₄, KOAc, quinuclidine, BiCl₃, Zn(CN)₂, Cu(OAc)₂, and NiCl₂.
 ACCESSION NUMBER: 1981:497404 CAPLUS
 DOCUMENT NUMBER: 95:97404
 TITLE: Urethanes and their use in isocyanate production
 INVENTOR(S): Koenig, Klaus; Findeisen, Kurt; Sundermann, Rudolf; Penninger, Stefan; Fauss, Rudolf
 PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
 SOURCE: Ger. Offen., 35 pp.
 CODEN: GWXXRX
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2943481	A1	19810507	DE 1979-2943481	19791027
EP 27940	A1	19810506	EP 1980-106196	19801011
EP 27940	B1	19830914		
EP 27940	B2	19861230		
R: AT, BE, CH, DE, FR, GB, IT, NL, SE				
AT 4640	E	19830915	AT 1980-106196	19801011
CA 1144562	A1	19830412	CA 1980-362772	19801020
BR 8006849	A	19810428	BR 1980-6849	19801024
ES 496232	A1	19810916	ES 1980-496232	19801024
ZA 8006546	A	19811125	ZA 1980-6546	19801024
JP 57114561	A2	19820716	JP 1980-148903	19801025
JP 03014819	B4	19910227		
AU 8063713	A1	19810430	AU 1980-63713	19801027
AU 538535	B2	19840816		

PRIORITY APPLN. INFO.: DE 1979-2943481 A 19791027
 EP 1980-106196 A 19801011

L20 ANSWER 132 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Alcs., aldehydes, and ketones reacted with aminating agents over Co-Cu and Ni-Cu mixts. containing one of Fe, Zn, and Zr. Thus, H₂NCH₂CH₂OH was treated with NH₃ and H (500 psig) over a Co-Cu-Fe mixture at .apprx.180° to give H₂NCH₂CH₂NH₂.
 ACCESSION NUMBER: 1981:156286 CAPLUS
 DOCUMENT NUMBER: 94:156286
 TITLE: Catalytic production of amines from alcohols, aldehydes, ketones, or their mixtures
 INVENTOR(S): Habermann, Clarence Edward
 PATENT ASSIGNEE(S): Dow Chemical Co., USA
 SOURCE: Eur. Pat. Appl., 22 pp.
 CODEN: EPXXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 17651	A1	19801029	EP 1979-100986	19790402
R: BE, DE, FR, GB, IT, NL, SE				

PRIORITY APPLN. INFO.: EP 1979-100986 19790402

L20 ANSWER 133 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB Amines were prepared by amination of aldehydes, ketones, alcs., or their mixts. over Ni, Co, Cu, Fe, Zn or Zr catalysts. Thus, a mixture of 20 g HZNCH2CH2OH, 75 g NH3 and 8 g catalysts containing Co 30, Cu 63, and Fe2O3 7 mol% in 500 psig H was heated 5 h at 180° to give 43 weight% HZNCH2CH2NH2 with 67% conversion.

ACCESSION NUMBER: 1981:102814 CAPLUS
DOCUMENT NUMBER: 94:102814
TITLE: Preparation of amines from alcohols, aldehydes, and ketones
PATENT ASSIGNEE(S): Dow Chemical Co., USA
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKKXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 55139342	A2	19801031	JP 1979-42076	19790409
PRIORITY APPLN. INFO.: JP 1979-42076 A 19790409				

L20 ANSWER 134 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB Aliphatic amines R1CH2N(R2)R3 (R1 = C7-23 aliphatic group; R2, R3 = H, C1-24 aliphatic group) are prepared by catalytic reductive aminolysis of R1CH2OH or

R1CHO with ammonia or primary or secondary amines R2R3NH. The catalysts are colloidal mixts. prepared by reduction of a 2-component system containing a Cu or Ag complex salt and a Group VIII element compound or Mn compound or Zn compound. Thus, a mixture containing 1-dodecanol, Cu acetylacetonate, stearic acid, and Ni stearate was reduced with H2, then treated with Me2NH and H2 to give 95.9% Me2N(CH2)11Me. The colloidal catalysts used gave higher yields than Raney Ni or Cu chromite.

ACCESSION NUMBER: 1981:83592 CAPLUS
DOCUMENT NUMBER: 94:83592
TITLE: Aliphatic amines
INVENTOR(S): Kimura, Hiroshi; Matsutani, Kazuhito; Tsutsumi, Shunichi
PATENT ASSIGNEE(S): Kao Soap Co., Ltd., Japan
SOURCE: Ger. Offen., 28 pp.
CODEN: GWXXRX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3005953	A1	19800911	DE 1980-3005953	19800216
JP 55111446	A2	19800828	JP 1979-19580	19790221
JP 59012106	B4	19840321		
US 4254060	A	19810303	US 1980-122405	19800219
GB 2046616	A	19801119	GB 1980-5740	19800220
PRIORITY APPLN. INFO.: JP 1979-19580 A 19790221				

L20 ANSWER 135 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB Homogeneous, colloidal catalyst systems, consisting of metal salts of organic acids, were developed for the amination of aliphatic alcs. with aliph amines; in general these were ternary and included a Cu or Ag salt (esp Cu2+ stearate) a Group VIII (including Mn and Zn) metal salt (especially Ni stearate) and an alkali or alkaline earth metal salt (especially Ba stearate). Thus, 300 g 1-dodecanol and Me2NH in the presence of 3 g cupric, 0.6 g Ni, and 0.6 g Ba stearates at 100° gave 96.1% dodecylidimethylamine.

ACCESSION NUMBER: 1980:41325 CAPLUS
DOCUMENT NUMBER: 92:41325
TITLE: Aliphatic amines
INVENTOR(S): Hoshino, Fumio; Kimura, Hiroshi; Matsutani, Kazuhito
PATENT ASSIGNEE(S): Kao Soap Co., Ltd., Japan
SOURCE: Ger. Offen., 45 pp.
CODEN: GWXXRX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2907869	A1	19790927	DE 1979-2907869	19790301
DE 2907869	C2	19880707		
JP 54125603	A2	19790929	JP 1978-30149	19780316
JP 57000849	B4	19820108		
GB 2017682	A	19791010	GB 1979-6736	19790226
GB 2017682	B2	19820825		
BR 7901449	A	19791009	BR 1979-1449	19790309
ES 478638	A1	19790701	ES 1979-478638	19790314
FR 2423475	A1	19791116	FR 1979-6462	19790314
FR 2423475	B1	19820730		
CA 1109491	A1	19810922	CA 1979-323556	19790316
PRIORITY APPLN. INFO.: JP 1978-30149 A 19780316				

L20 ANSWER 136 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB Amines were prepared by the ammonolysis of alcs., aldehydes and ketones over catalysts containing (mol. % on an oxide-free basis) 20-90% Co, 8-72% Cu, and 1-16% of 1 or more of Fe, Zn or Zr. Thus, HOCH2CH2NH2 20 and NH3 75 g, with 500 psig H and 8 g catalyst (30% Co, 63% Cu, 7% Fe) heated 5 h at 180° gave 67% conversion with 43% HZNCH2CH2NH2 in the product mix.

ACCESSION NUMBER: 1979:438905 CAPLUS
DOCUMENT NUMBER: 91:38905
TITLE: Amines from alcohols, aldehydes, ketones and mixtures
INVENTOR(S): Habermann, Clarence E.
PATENT ASSIGNEE(S): Dow Chemical Co., USA
SOURCE: U.S., 6 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4153581	A	19790508	US 1977-829758	19770901
CA 1126248	A1	19820622	CA 1979-324955	19790405
PRIORITY APPLN. INFO.: US 1977-829758 19770901				

L20 ANSWER 137 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The reaction of alcs., aldehydes, ketones, and their mixts. with
 aminating agents was catalyzed by three-component systems containing Ni,
 Cu, and Fe, Zn, Zr, and their mixts., and the resp.
 amines were obtained. An autoclave was charged with H₂NCH₂CH₂OH, NH₃, and
 a catalyst containing Ni 30, Cu 63, and Fe203 7 mol %, and
 the autoclave was heated to .apprx.180° and kept at that temperature 5 h
 to yield H₂NCH₂CH₂NH₂.

ACCESSION NUMBER: 1979:419885 CAPLUS
 DOCUMENT NUMBER: 91:19885
 TITLE: Amines from alcohols, aldehydes, ketones and their
 mixtures
 INVENTOR(S): Habermann, Clarence E.
 PATENT ASSIGNEE(S): Dow Chemical Co., USA
 SOURCE: U.S., 5 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4152353	A	19790501	US 1977-828464	19770829
PRIORITY APPLN. INFO.: US 1977-828464 A 19770829				

L20 ANSWER 138 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB C3-10 amines were prepared from C3-10 alcs. and/or aldehydes and
 NH₃ by catalytic hydrogenation at 150-250° and 10-60 atmospheric The
 catalysts, prepared by alkaline treatment of alloys containing Ni 40-8, Al
 40-50, Cr 1-6, and/or Fe 1-6, and/or Si 1-2 weight% to the point of
 .apprx.50% of the Al content, followed by treatment with promoters, such
 as Ni, Pd, Co, Mg, Cr, an Cu, were stable for >1000 h. Thus, an
 alloy containing Al 47, Ni 48, Cu 3, and Si 2% was treated with 3%
 aqueous NaOH at 40-5° until the alloy contd. 23% Al, the alloy was
 treated with 1% Co(OAc)₂ and 1% Zn(OAc)₂ solns. for 12 h, washed
 until pH 10, dried in a mixt. of 80% H and 20% N, heated at 60 and
 100° in the same gas, heated at a rate of 4-5°/h to
 210° and kept at constant temperature for 2 h to give 3 mL catalyst
 . A mixture (90 mL/h) of 1.5 L BuOH and 4 L liquid NH₃ was passed through
 the catalyst together with a 4:1 mixture of H and N at 210° and
 60 atm to give a mixture of BuNH₂ 73.4, Bu₂NH 24.1, and Bu₃N 1.4%. The
 catalyst had a constant activity after 1000 h.

ACCESSION NUMBER: 1978:579532 CAPLUS
 DOCUMENT NUMBER: 89:179532
 TITLE: Amines
 INVENTOR(S): Csaszar, Mrs. Erno; Horvath, Laszlo; Hesser, Mrs.
 Odon; Klisgergely, Lajos; Jesztl, Mrs. Janos; Kincses,
 Gyula; Nagy, Csaba; Szeller, Bela; Schneider, Mrs.
 Janos; Szentgyorgyvari, Eva
 Peti Nitrogenmuvek, Hung.
 PATENT ASSIGNEE(S): Hung. Teljes, 19 pp.
 SOURCE: CODEN: HUXXBU
 DOCUMENT TYPE: Patent
 LANGUAGE: Hungarian
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
HU 14908	O	19780527	HU 1976-PE972	19760223
HU 172700	P	19781128		
PRIORITY APPLN. INFO.: HU 1976-PE972 A 19760223				

L20 ANSWER 139 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Amination catalysts combining Cu or its oxide with
 Zn and Cr or their oxides have a synergistic effect to give higher
 selectivity and catalyst stability in reactions of methylamines
 with alcs. or aldehydes. Thus, passing 3:1 MeNH₂-1-dodecanol in
 a H₂ stream continuously over a catalyst of 5% Cu, 4%
 Zn, and 1% Cr on Al₂O₃ at 210° gave 98% alc.
 conversion and selectivities of 93.2% Me(CH₂)₁₁NHMe and 5.1% C₂₄ and C₂₅
 amines. Extensive data obtained under other conditions were tabulated.

ACCESSION NUMBER: 1978:508097 CAPLUS
 DOCUMENT NUMBER: 89:108097
 TITLE: Dodecylamines
 INVENTOR(S): Slaugh, Lynn Henry
 PATENT ASSIGNEE(S): Shell Internationale Research Maatschappij B. V.,
 Neth.
 SOURCE: Ger. Offen., 23 pp.
 CODEN: GWXXBX
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2749066	A1	19780518	DE 1977-2749066	19771102
AU 7729705	A1	19780426	AU 1977-29705	19771014
AU 513156	B2	19801120		
BE 860236	A2	19780428	BE 1977-8479	19771028
NL 7712066	A	19780508	NL 1977-12066	19771102
JP 53059603	A2	19780529	JP 1977-130914	19771102
JP 60049178	B4	19851031		
FR 2370030	A1	19780602	FR 1977-32868	19771102
FR 2370030	B1	19800118		
GB 1554516	A	19791024	GB 1977-45583	19771102
PRIORITY APPLN. INFO.: US 1976-738814 A 19761104				

L20 ANSWER 140 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 GI For diagram(s), see printed CA issue.
 AB The reaction of cycloalkenes 1 [R groups (same or different) = H, alkyl, n
 = 2, 4, 5, 6; R1 = alkyl, aralkyl] with CO, monohydric saturated aliphatic,
 alicyclic, or aralkyl alcs., and O at 500-3000 psig and
 50-200° to give oxalate esters was catalyzed by mixts. of a) Pd,
 Pt, Rh, Cd, Co, Zn, and Cu salts, b) aliphatic,
 cycloaliph., aromatic, or heterocyclic amines or NH₃, c) cupric or ferric
 salts, and d) ammonium (substituted or unsubstituted) salts. Thus, MeOH
 and 1-methoxycyclohexene were treated with CO and O over Et₃N, H₂SO₄,
 Pd₂, Lil, and CuSO₄ at 100° to give MeO₂CCO₂Me.

ACCESSION NUMBER: 1978:120623 CAPLUS
 DOCUMENT NUMBER: 88:120623
 TITLE: Oxalate esters from carbon monoxide, alcohols, and
 alkoxy-cycloalkenes
 INVENTOR(S): Zehner, Lee R.
 PATENT ASSIGNEE(S): Atlantic Richfield Co., USA
 SOURCE: U.S., 8 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4069388	A	19780117	US 1976-734799	19761022
CA 1096880	A1	19810303	CA 1977-270671	19770128
CA 1091692	A1	19801216	CA 1977-284359	19770809
NL 7709193	A	19780425	NL 1977-9193	19770819
JP 53053612	A2	19780516	JP 1977-108688	19770909
JP 57005209	B4	19820129		
ES 462298	A1	19780516	ES 1977-462298	19770912
BE 859919	A1	19780419	BE 1977-181901	19771019
FR 2368461	A1	19780519	FR 1977-31436	19771019
DE 2747363	A1	19780427	DE 1977-2747363	19771021
DE 2747363	C2	19820923		
GB 1572145	A	19800723	GB 1977-43963	19771021
PRIORITY APPLN. INFO.: US 1976-734799 A5 19761022				

L20 ANSWER 141 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The oxidative carbonylation of R2C:CR1OR2 (R and R1 are H, alkyl, cycloalkyl, aralkyl, aryl; R2 = alkyl, aralkyl) mixed with the resp. R2OH at 500-3000 psig and 50-200° over catalysts prepared from (a) a Pt, Pd, Rh, Cd, Co, Zn, or Cu salt, (b) an amine or NH3, (c) a cupric or ferric salt, and (d) an ammonium salt (formed in situ by the addition of acid) gave the resp. R2O2CCO2R2. A BuOH-BUOCH:CH2 mixture was treated with CO and O at 1800 psig and 90° over a mixture containing Bu3N, H2SO4, PdSO4, and CuSO4 to give BuO2CCO2Bu.
 ACCESSION NUMBER: 1978:89145 CAPLUS
 DOCUMENT NUMBER: 88:89145
 TITLE: Oxalate esters from carbon monoxide and an enol ether
 INVENTOR(S): Zehner, Lee R.
 PATENT ASSIGNEE(S): Atlantic Richfield Co., USA
 SOURCE: U.S., 7 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION: .

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4065490	A	19771227	US 1976-669376	19760322
PRIORITY APPLN. INFO.:			US 1976-669376	A 19760322

L20 ANSWER 142 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Mesityl oxide platinum dichloride (I) [58601-82-4]-catalyzed reaction of an allylamine with an alkoxy silane gave β -aminoethylsilanes useful for promoting adhesion of PVC [9002-86-2] to Cu [7440-50-8], Zn [7440-66-6], Al [7429-90-5], and Sn [7440-31-5], inhibiting corrosion of Cu, and sizing glass fibers. Thus, 492 g triethoxysilane [998-30-1] was added during 2 hr to 190 g refluxing allylamine [107-11-9] containing 2 ml of a 0.1M solution of I in Me2CO, the mixture was refluxed until a sump temperature of 135°, giving, after fractional distillation in vacuo 130 g of a composition containing 95% α -methyl- β -aminoethyltriethoxysilane (II) [36957-84-3]. Treatment of degreased and pickled Cu strips with 10% alc. solution of II followed by drying 1 hr at 130° gave a hard firmly adhering coating. After 3 hr in an oven at 150° the above treated strips retained their appearance whereas untreated strips showed marked discoloration.
 ACCESSION NUMBER: 1976:123587 CAPLUS
 DOCUMENT NUMBER: 84:123587
 TITLE: Aminosilanes
 INVENTOR(S): Amort, Juergen; Nestler, Heinz; Gerhardt, Peter; Seiler, Claus D.; Vahlensieck, Hans J.
 PATENT ASSIGNEE(S): Dynamit Nobel A.-G., Fed. Rep. Ger.
 SOURCE: Brit., 6 pp.
 CODEN: BRGXAA
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION: .

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1409483	A	19751008	GB 1973-12894	19730316
PRIORITY APPLN. INFO.:			GB 1973-12894	A 19730316

L20 ANSWER 143 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Higher (C8 to C20) aliphatic amines were prepared by vapor-phase reaction of aliphatic secondary alcs. or ketones with NH3-H at ordinary pressures in the presence of Cu, Zn, Cr, Mo, Co, or Ni and their oxides. Thus, 50 weight % kieselguhr was added to aqueous Co and Cu nitrates (8:2 CoCu), aqueous NaOH added, and the solid product dried and calcined to form a catalyst of Co oxide-Cu oxide. The catalyst (80 ml) was treated with H4 hr at 250-300° and 8 ml/hr sec-tetradecanol fed at 0.1 ml/hr-ml together with 20.7 l./hr NH3 and 6 l./hr H at 180° for 10 hr to give a mixture containing sec-tetradecanol 1.8, tetradecanone 2.5, tetradecylamine 90.4, ditetradecylamine 5.2, and tritetradecylamine 0.1 mole %.
 ACCESSION NUMBER: 1974:59430 CAPLUS
 DOCUMENT NUMBER: 80:59430
 TITLE: Higher aliphatic amines
 INVENTOR(S): Umemura, Sumio; Takamitsu, Nagaaki; Hanamoto, Toshikazu; Ito, Yukikatsu
 PATENT ASSIGNEE(S): Ube Industries, Ltd.
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
 CODEN: JKXXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION: .

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 48085511	A2	19731113	JP 1972-17658	19720222
PRIORITY APPLN. INFO.:			JP 1972-17658	A 19720222

L20 ANSWER 144 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Impregnation of Kapron fabric with an aqueous emulsion containing GKZh-94*poly(ethylhydrosiloxane) (I) 0.6%, Solvar [9002-89-5] 0.01%, and a catalytic amount of copper acetate monohydrate [6046-93-1], lead diacetate [301-04-2], zinc acetate [557-34-6], or lead diacetate trihydrate-bis(triethanolamine) complex, wringing, and drying at 100.deg. gave waterproof fabric, suitable for use in shoe manufacturing I was more effective than poly(methylhydrosiloxane). I forms a porous film on the fabric surface which does not impede gas permeation through the fabric.
 ACCESSION NUMBER: 1972:114654 CAPLUS
 DOCUMENT NUMBER: 76:114654
 TITLE: Use of organosilicon compounds for waterproofing
 AUTHOR(S): Kucher, I. E.
 CORPORATE SOURCE: USSR
 SOURCE: Khimicheskaya Tekhnologiya (Kiev) (1971), (5), 47-8
 CODEN: KHMTA6; ISSN: 0368-556X
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 145 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The title conversion was carried out by treating in the vapor phase, at 100-250°/10-250 atm, an alc. with a primary or secondary amine in the presence of H or H₂O and a catalyst containing 70-95 of a Co + Ni mixture and 5-30, preferably 15 weight % Cu, the Co-Ni weight ratio being between 4:1 and 1:4. Eventually may be added Mn, Cr, Zn, V, Ag, Na, K, Ca, Ba, H₃PO₄, or B(OH)₃ (1-20% based on the metal catalyst). Thus, into a vertical tube was charged a catalyst containing 10 Co₂O₃, 10 NiO, 4 CuO and 0.52 weight % H₃PO₄. After reducing the catalyst with H at 250°, there was introduced, at the top, poly(propylene glycol) (PM 1400) and liquid NH₃ at resp., 100 weight parts and 350 ml. per hr, while maintaining 180° and 300 atmospheric H pressure to give a product of amine index 76, corresponding to 95% conversion.

ACCESSION NUMBER: 1972:99094 CAPLUS
 DOCUMENT NUMBER: 76:99094
 TITLE: Catalytic conversion of alcohols to amines
 PATENT ASSIGNEE(S): Badische Anilin- & Soda-Fabrik AG
 SOURCE: Fr., 9 pp.
 CODEN: FROKAX
 DOCUMENT TYPE: Patent
 LANGUAGE: French
 FAMILY ACC. NUM. COUNT: 2
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2066157	A5	19710806	FR 1970-37604	19701019
DE 1953263	A	19720217	DE 1969-1953263	19691023
DE 1953263	C2	19870122		

PRIORITY APPLN. INFO.: DE 1969-1953263 A 19691023

L20 ANSWER 146 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Vinyl chloride was polymerized in the presence of a bidentate ligand containing O-N, N-N, and O-O donor atoms, a transition metal salt, and a halogen-containing compound. Aminoalc. derivs. were the most effective bidentate ligand studied. The effect of the individual metal salts on the polymerization rate (Rp) in the initiation system 1-amino-2-propanol (I) [78-96-6]-carbon tetrachloride [56-23-5] was in the order: Cu(II) >> Mn(II), Fe(III), Pb(II), Ni(II), Cr(III), Co(II), Zn(II), > Fe(II). Rp depended on the I and Cu(OAc)₂ concentration according to the equation $R_p = k [\text{amino alc.}]^{1.85} [\text{Cu(OAc)}_2]^{0.51}$. Rp was not affected by the addition of poly(vinyl chloride) [9002-86-2]. The Rp temperature dependence at 0-30 deg. corresponded to the Arrhenius equation.

ACCESSION NUMBER: 1972:46564 CAPLUS
 DOCUMENT NUMBER: 76:46564
 TITLE: Radical reactions initiated by chelate complexes of transition metals. IV. System bidentate ligand-salt of the transition metal-halogen-containing compound as initiator of vinyl chloride polymerization
 AUTHOR(S): Barton, J.; Manasek, Z.; Lazar, M.
 CORPORATE SOURCE: Inst. Polym., Slovak Acad. Sci., Bratislava, Czech.
 SOURCE: Chemické Zvesti (1971), 25(4), 292-9
 CODEN: CHZVAN; ISSN: 0366-6352
 DOCUMENT TYPE: Journal
 LANGUAGE: English

L20 ANSWER 147 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Primary amines, useful for the manufacture of emulsifiers, plant protecting agents, vulcanization accelerators, and pharmaceuticals, were prepared by reaction of the corresponding alcs. with NH₃ in the presence of a Co catalyst containing P205 and B2O3. Thus, HOCH₂CH₂NH₂ (I), H₂O, and NH₃ were introduced at 195° and 300 atm into a high-pressure tube filled with a catalyst containing oxides of Co 20, Mn 0.7, Zn 1, Cu 4, Cr 1.8, Ag 1, P 1, and B 0.25%, which was hydrogenated at 250°, to give 83% (based on transformed I) H₂NCH₂CH₂NH₂. Similarly prepared were, e.g. morpholine, cyclohexylamine, and n-C₁₈H₃₇NH₂.

ACCESSION NUMBER: 1971:436071 CAPLUS
 DOCUMENT NUMBER: 75:36071
 TITLE: Amines by dehydroamination of alcohols using cobalt catalysts
 INVENTOR(S): Corr, Hubert; Hoffmann, Herwig; Toussaint, Herbert; Winderl, Siegfried; Boettger, Guenter
 PATENT ASSIGNEE(S): Badische Anilin- & Soda-Fabrik AG
 SOURCE: Ger. Offen., 11 pp.
 CODEN: GWXEXX
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1950604	A	19710422	DE 1969-1950604	19691008
NL 7014460	A	19710414	NL 1970-14460	19701001
FR 2065046	A5	19710723	FR 1970-35984	19701006

PRIORITY APPLN. INFO.: DE 1969-1950604 A 19691008

L20 ANSWER 148 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Textiles, containing at least 25% cellulosic fibers, were treated for improved abrasion resistance, wrinkle resistance, crease retention and durable press properties. The fabrics were treated with N-methylol or N-methoxymethyl crosslinking agents in the presence of a polymerization catalyst and later impregnated with an acid latent catalyst and dried. The second cure could be postponed for storage \leq 12 months. Thus, a sateen cotton fabric was padded with an aqueous solution containing methylated methyloated melamine, dimethylol-4,5-dihydroxyethyleneurea, zirconium acetate, AcOH and an alkylaryl polyether alc. wetting agent. The fabric was dried 3 min at 180°F, cured 3 min at 320°F, washed and dried. The fabric was padded with an aqueous emulsion containing Zn(NO₃)₂·6H₂O and an emulsified mixture of polyethylene and polypropylene. The fabric was dried and cured. Significant improvements were noted in the wrinkle recovery values. The treatment imparted good wrinkle resistance without introducing loss in tear strength, which is common to this type treatment. Similarly used were Al, Pb, Mn, Cu, Mg and Zn acetates. Also used in the 2nd step were MgCl, NH₄Cl, Zn fluoborate, (NH₄)₂SO₄, and AlCl₃.

ACCESSION NUMBER: 1970:489103 CAPLUS
 DOCUMENT NUMBER: 73:89103
 TITLE: Abrasion-resistant durable-press cellulosic textiles
 INVENTOR(S): Reeves, Wilson A.; Hamalainen, Carl; St. Mard, Hubert H.; Cooper, Albert S., Jr.
 PATENT ASSIGNEE(S): United States Dept. of Agriculture
 SOURCE: U.S., 10 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3526474	A	19700901	US 1966-584068	19660930

PRIORITY APPLN. INFO.: US 1966-584068 A 19660930

L20 ANSWER 149 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Catalytic activities for decomposition of H₂O₂, NH₂NH₂ and for oxidation of pyrogallol, catechol, and ascorbic acid were investigated on intramol. complexes prepared from transition metal ions Ni, Cu, Fe, Zn, Pb, Mg, Co, Cd, and Schiff bases from salicylaldehyde (II) with o-, p-, m-NO₂C₆H₄NH₂, o-, p-, m-NH₂C₆H₄OH, o-, p-, m-(NH₂)₂C₆H₄, NH₂NHPh, and on the complexes prepared from transition metal ions and azomethines from salicylaldehyde with ethylenediamine, NH₂Ph, NH₂(CH₂)₆NH₂, o-NH₂C₆H₄Ph. The activities were characterized by the volume of gas produced in 15-min. reactions at 50 mg. reactants in 50 cm.³ solution from H₂O₂-alc. The order of activity of chelate complexes from salicylaldehyde and p-NH₂C₆H₄OH with transition metals was: Cu > Fe > Mg > Co > Ni > Zn.
 1969:464600 CAPLUS
 DOCUMENT NUMBER: 71:64600
 TITLE: Catalytic activity of azomethine-transition metal complexes
 AUTHOR(S): Movchan, L. A.; Kudryavtsev, A. S.; Savich, I. A.
 CORPORATE SOURCE: Mosk. Gos. Univ. im. Lomonosova, Moscow, USSR
 SOURCE: Zhurnal Fizicheskoi Khimii (1969), 43(6), 1584-6
 CODEN: ZFKH99; ISSN: 0044-4537
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 150 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Initiator systems consisting of an oxidizing agent, a Ti, Fe, V, Al, Sn, Mn, Cr, Co, Cu, Zn, or Bi chelating agent, and an electron donor were used to polymerize α,β -unsatd. monomers. The initiators produced polymers which had thermoch. properties similar to those of polymers obtained at lower temps. with other initiating systems. Thus, to 400 cc. H₂O, 1 g. poly(vinyl alc.) having a saponification index of 125, 2 g. lauroyl peroxide (I), 0.650 g. vanadyl acetylacetonate (II), and 0.200 g. dialaurylamine at 16° under N was added 100 g. vinyl chloride (III) and the mixture kept 20 hrs. at 16° to give 60 g. poly(vinyl chloride) having viscosity index (5 g./l. in cyclohexanone at 25°) 282 cc./g. The polymer had properties similar to those of polymers obtained at -10° using trichloroacetyl peroxide catalyst. Similar results were achieved using a Bz₂O₂, ferrous acetylacetonate (IV), and pyridine catalyst with poly(vinylpyrrolidone) (V) suspending agent. An increase in the amount of electron donor beyond certain values resulted in a lowering of the polymer yield and the viscosity index, and caused some crosslinking of the polymer. For a given monomer, the polymer yield was higher with primary amines than with tertiary amines. The following polymers of III were carried out (oxidizing agent, chelate, and electron donor given): I, II, MeNH₂; I, II, Et₃N; I, II, Et₂NH; I, vanadyl acetylacetonate, Ph₂NH; I, vanadyl 2-acetylacetonate, Ph₂NH; I, vanadyl heptanedionate, Ph₂NH; I, II, NH₄OH; 2,4-dichlorobenzoyl peroxide (VI), II, NH₄OH; cumene hydroperoxide, II, NH₄OH; VI, vanadyl propionylacetaldehyde (VII), NH₄OH; VI, vanadyl benzoylacetaldehyde (VIII), NH₄OH; VI, dichlorotitanium acetylacetonate, NH₄OH; VI, diethyltin acetylacetonate, NH₄OH; VI, cupric acetylacetonate, NH₄OH; VI, vanadyl salicylaldehyde (IX), NH₄OH; VI, vanadyl bis(acetoacetamide) (X), NH₄OH; VI, vanadyl bis(acetoacetanilide) (XI), NH₄OH; VI, vanadyl thioglycolate (XII), NH₄OH; VI, vanadyl anthranilate (XIII), NH₄OH; VI, II, acetamide; VI, II, Et₂O; VI, II, dioxane; VI, II, urea; VI, zinc acetylacetonate, Et₂O; I, II, hydrazine; VI, ethoxylaluminum acetylacetonate, NH₄OH; di-tert-butyl peroxide, manganous acetylacetonate, NH₄OH; VI, titanil acetylacetonate (XIV), NH₄OH; VI, ethyltitanil bis(acetoacetate), NH₄OH; 3-isopropyl-3-butyloxazirane, II, NH₄OH. III was emulsion polymerized by replacing the V with Na dodecylbenzenesulfonate. A catalyst system of KBrO₃, II, and Et₂O gave 94% conversion and a polymer having viscosity 285 cc./g. The following solution polymers. of III were carried out (oxidizing agent, solvent, chelating agent, and electron donor given): KBrO₃, MeOH, II, cyclohexylamine (XV); I, hexane, vanadyl 3-chloroacetylacetonate, XVI; I, MeOH, II, isopropylamine; I, MeOH, II, NH₄OH. The following similar polymers were carried out (monomer, mode of polymerization, reaction medium, oxidizing agent, chelating agent, and electron donor given): vinyl acetate (XVI), suspension, H₂O, VI, II, NH₄OH; XVI, suspension, H₂O, VI, II, Et₂O; XVI, suspension, H₂O, VI, II, Bu₂O; XVI, suspension, H₂O, VI, II, tetrahydrofuran (XVII); XVI, emulsion, H₂O, KBrO₃, II, XVII; XVI, suspension, H₂O, I, XIV, NH₄OH; XVI, suspension, H₂O, VI, IX, NH₄OH; XVI, suspension, H₂O, VI, X, NH₄OH; XVI, suspension, H₂O, VI, XI, NH₄OH; XVI, suspension, H₂O, VI, XII, NH₄OH; XVI, suspension, H₂O, VI, XIII, NH₄OH; vinyl benzoate, solution, MeOH, I, II, NH₄OH; acrylonitrile (XVIII), solution, HCONMe₂, I, II, NH₄OH; XVIII, suspension, H₂O, VI, vanadyl α -formylcyclohexanone, NH₄OH; XVIII, suspension, H₂O, VI, VII, NH₄OH; XVIII, suspension, H₂O, VI, VIII, NH₄OH; XVIII, aq., Bz₂O₂, IV, piperidine; vinylidene chloride, suspension, H₂O, VI, II, NH₄OH; but-3-en-2-one, suspension, H₂O, VI, II, NH₄OH; XVI, emulsion, H₂O, KClO₄, II, urea.
 1969:38282 CAPLUS

L20 ANSWER 150 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 DOCUMENT NUMBER: 70:38282
 TITLE: Polymerization initiator for olefinic monomers
 PATENT ASSIGNEE(S): Rhone-Poulenc S. A.
 SOURCE: Brit., 22 pp.
 CODEN: BRXXAA
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1136326		19681211		
PRIORITY APPLN. INFO.:			FR	19660513

L20 ANSWER 151 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Catalyst systems used for polymers. at -30 to 100° are prepared from an organic or mineral oxidant; a chelated derivative of a bidentate ligand with V, Ti, Fe, Sn, Zn, or Mn; and an electron donor, such as NH₄OH or BuNH₂. Thus, a mixture of poly(vinyl alc.) 1, H₂O 400, lauroyl peroxide (I) 2, vanadyl acetylacetonate (II) 0.650, and dialaurylamine (III) 0.2 g. was placed in an autoclave at 16°, then 100 g. vinyl chloride was introduced. After 20 hrs. at 16°, the polymer was filtered and dried in vacuo to give 60 g. poly(vinyl chloride) (IV) with a viscosity index of 282 cc./g. (0.5% cyclohexane at 25°). IV was prepared similarly by use of NH₄OH instead of III. Bz₂O₂, 2,4-dichlorobenzoyl peroxide, cumene hydroperoxide, KBrO₄, or 3-butyl-3-isopropylloxazirane were used instead of I in the catalyst systems used. Titanil acetylacetonate, vanadyl salicylaldehyde, bis(acetylacetonate), thioglycolate, heptanedionate, anthranilate, α -formyl cyclohexanone, propionyl acetaldehyde, and benzoylacetaldehyde, and Fe, Cu, Zn, Mn, and ethoxylaluminum acetylacetonate were used as the chelates in the catalyst. Ph₂NH, BuNH₂, Bu₂NH, MeNH₂, Et₃N, Et₂NH, cyclohexylamine, AcNH₂, ethyleneoxide, iso-PrNH₂, or N₂H₄ were used as the electron donors. Polymerization conditions are given for vinyl acetate, vinyl benzoate, acrylonitrile, vinylidene chloride, 3-butene-2-one, C₃H₆, C₂H₄, and vinyl chlorides. Poly(vinylpyrrolidone), Na dodecylbenzenesulfonate, and polyethylene glycol stearate were used as emulsifiers in the emulsion.
 1968:105650 CAPLUS
 DOCUMENT NUMBER: 68:105650
 TITLE: Initiation of polymerization of monomers containing olefinic double bonds
 INVENTOR(S): Chabert, Henri; Chapurlat, Robert; Gigu, Claude; Rnaud, Michel
 PATENT ASSIGNEE(S): Societe des usines chimiques de Rhone-Poulenc
 SOURCE: Fr., 17 pp.
 CODEN: FRXXAX
 DOCUMENT TYPE: Patent
 LANGUAGE: French
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1492940		19670825	FR	19660513
DE 1745333			DE	
US 3860568		19750000	US	

L20 ANSWER 152 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Flexible, light-stable polyurethane foams are prepared by simultaneously mixing a hydroxylated polyester or polyether, an aliphatic diisocyanate, a polyfunctional OH compound, H₂O, a metallic catalyst, and an optional stabilizer. Thus, a trifunctional polyester having a mol. weight of 3000 and OH index 6050, Fomrez 77-86 stabilizer 1.0, and N,N,N',N'-tetrahydroxyethoxyethylenediamine 2.0 g., and 17 ml. hexamethylene diisocyanate were premixed for 60 sec., a 5% aqueous solution containing 1.5 ml. triethylenediamine and 1.0 ml. stannous octoate added, and mixed for 60 sec., the composition foamed to a maximum volume of 1800 ml. in 6.5 min., the foam heated at 100°, and the numerous closed cells broken by compression. A sample exposed under glass to daylight for 6 months remained unchanged. Similarly used were glycerol or triethanolamine; bis(4-isocyanatocyclohexyl)methane, HCN/diethylene glycol divinyl ether, bis(2-isocyanatoethyl)carbonate, or bis(2-isocyanato ethyl) fumarate; diethylene glycol-adipic acid polyester, Voranol CP-2000, or poly(propylene glycol ether); Cd octoate, Zn octoate, Cu acetylacetonate, or Fe(III) acetylacetonate; and- (or) Dow Corning DC-199 stabilizer.

ACCESSION NUMBER: 1968:3566 CAPLUS
 DOCUMENT NUMBER: 68:3566
 TITLE: Light-stable, flexible polyurethane foams
 INVENTOR(S): Wilson, Christopher Lumley
 PATENT ASSIGNEE(S): Johnson and Johnson
 SOURCE: Fr., 12 pp.
 CODEN: FRXOAX
 DOCUMENT TYPE: Patent
 LANGUAGE: French
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1481815		19670519		
DE 1694417			DE	
GB 1150425			GB	
PRIORITY APPLN. INFO.:			US	19650602

L20 ANSWER 153 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 Moscov, USSR
 SOURCE: Khimiya Geterotsiklicheskikh Soedinenii (1967), (3), 522-6
 CODEN: KGSSAQ; ISSN: 0132-6244
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 153 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 GI For diagram(s), see printed CA issue.
 AB 3,4-(MeO)C₆H₃(CH₂)₂NH₂ (10 g.) and 9.98 g. 4-MeOC₆H₄CH₂CO₂H was heated 1 hr. at 190-5° in a N stream and the mixture ground with Et₂O to give 89.2% 3,4-(MeO)C₆H₃(CH₂)₂NHCO₂CH₂CO₂H (I), m. 138.5-9° (alc.). 3,4-(MeO)(PhCH₂O)C₆H₃(CH₂)₂NH₂ (4.6 g.), 3.2 g. 4-MeOC₆H₄CH₂CO₂H, and 1.2 cc. CSH₅N was heated 5 hrs. at 185-90°, CSH₅N removed in vacuo, the residue dissolved in CHCl₃, and washed with dilute HCl and H₂O to give 84.5% 3,4-(MeO)(PhCH₂O)C₆H₃(CH₂)₂NHCO₂CH₂CO₂H (II), m. 114-15° (alc.). EtONa (from 0.962 g. Na and 34 cc. alc.) was added to 13.5 g I in 100 cc. EtOH, 5.24 g. PhCH₂Cl added after 15 min., and the mixture refluxed 5 hrs. to give 75.5% II (10.15 g.), 100 cc. PhMe, and 13.86 cc. POCl₃ was refluxed 1.5 hrs. in a N stream to give 98.2% 1-(4-methoxybenzyl)-6-methoxy-7-benzoyloxy-3,4-dihydroisquinoline (III) hydrochloride, m. 200-1° (decomposition) (alc.); picrate m. 195-6° (AcOH); methiodide m. 167-7.5° (EtOH); methochloride (IV) m. 49-50°. IV (1 g.) in 20 cc. EtOH was hydrogenated over 0.5 g. PdO₂ for 30 min. to give 89.9% V hydrochloride (VI), m. 48-9.5°. IV (6 g.) in 250 cc. AcOH and 69 cc. H₂O was refluxed with 46.5 g. Zn dust for 2.5 hrs., Zn separated, the filtrate cooled, neutralized with NH₃, and extracted with CHCl₃, and the CHCl₃ layer extracted with dilute (1:3) HCl to give 90.4% VII. VI (0.88 g.) in 30 cc. MeOH was hydrogenated over 0.3 g. Pd-black 5 hrs., the product dissolved in 10% NaOH, unchanged V extracted with Et₂O, and the aqueous layer worked up to give 68.4% VII.HCl, m. 160-9° (decomposition). VII.HCl (1.65 g.), 2.8 g. 3,4-Br(MeO)C₆H₃CH₂CO₂H, 0.66 g. fine powdered anhydrous K₂CO₃, 1 cc. anhydrous CSH₅N, and 0.1 g. precipitated Cu

Catalyst (VIII) was heated 10 min. at 160°, 20 min. at 170°, and 1.5 hrs. at 185°, the mixture dissolved in 25 cc. H₂O and 300 cc. CHCl₃ and the organic layer yielded 53.4% IX, m. 123-30° (ground with Et₂O); HCl salt m. 138-42°. VII (1.2 g.) was dissolved in MeOH (from 12 mg. K and 11 cc. MeOH), MeOH evaporated, the residue dried at 100°, ground, heated with 1.06 g. VIII and 1.2 g. 3,4-Br(HO)C₆H₃CH₂CO₂H for 1 hr. with stirring at 185-90° in a N stream, and the product dissolved in CSH₅N and chromatographed on Al₂O₃ (activity 4) to give 33.9% X; HCl salt m. 80-97° (decomposition). Similarly, 41.2% Me ester (HCl salt m. 220-4°) was obtained. IX (0.52 g.), 0.4 g. homoveratrylamine, and 0.5 cc. anhydrous CSH₅N was heated in the N atmosphere for 4 hrs. at 182-5° to give 85.1% 1-(4-methoxybenzyl)-6-methoxy-7-[2-methoxy-5-[β-(3,4-dimethoxyphenyl)ethylcarbamidomethyl]phenoxy]-N-methyl-1,2,3,4-tetrahydroisquinoline (XI); HCl salt m. 101-3°. XII (64.4%) (HCl salt m. 163-8°) was obtained analogously to III. XIII (72%), m. 98-100°, was obtained similarly to V. XIII (0.54 g.), 5 cc. 85% HCO₂H, and 7.4 cc. 34% HCHO was heated 5 hrs. at 95-7°, the mixture treated with 60 cc. 5% NaOH and 300 cc. Et₂O; the organic layer contained 75.6% XIV.HCl, m. 97-9°. β-(3-Methoxy-4-[2-acetoxy-5-[β-(3-methoxy-4-acetoxyphenyl)ethylcarbamidomethyl]phenoxy]phenyl)ethylamide of 4-benzoyloxyacetic acid gave, by cyclization with POCl₃ in PhMe, XV, m. 88-91°, HCl salt m. 160-1.5°.

ACCESSION NUMBER: 1968:3039 CAPLUS
 DOCUMENT NUMBER: 68:3039
 TITLE: Synthetic studies in the curare alkaloid area. XIX.
 AUTHOR(S): Synthesis of the dimethyl ester of racemic lianinine
 Volkova, L. V.; Kosmachova, V. E.; Voronin, V. G.;
 Tolkachov, O. N.; Preobrazhenskii, N. A.
 CORPORATE SOURCE: Mosk. Inst. Tonkoi Khim. Tekhnol. im. Lomonosova,

L20 ANSWER 154 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Economical polyester resins having improved mech., chemical, elec., and thermal properties are described. The resins are adaptable for use in surface coatings, laminates, films, and wire enamels, e.g., insulation for elec. conductors, and slot insulation in elec. apparatus. The polyester resins are prepared from isocyanuric derivs. (I). The polyesters may be derivs. of polycarboxylic acids, glycols, or polyols, cross-linked with conventional curing or modifying agents with or without metal catalysts, and prepared from fatty acids and (or) oils. The resins prepared from a phthalic acid or ester (II), especially orthophthalic and terephthalic esters, ethylene glycol (III), and tris(hydroxyethyl) isocyanurate (IV). Cresylic acid (V) may also be used during esterification. A superior product is obtained by transesterifying II with IV prior to esterifying its product with III in the presence of V. Thus, di-Me phthalate (VI) 43.45, III 27.55, and IV 29.0% were heated to 300°F. under CO₂, 0.1% PbO esterification catalyst added, and the mixture heated 6 hrs. as alc. was distilled and the temperature rose to 430°F., resulting in a resin with 31% viscosity. Another resin was prepared as above until 37% viscosity was reached, V added to yield a solution having 70% solids, and esterification continued at 430°F. until 34% viscosity was reached. A third polyester resin was prepared by a 2-step process in which 43.35% VI and 29.10% IV were heated to 300°F. under CO₂, 0.1% PbO added, the mixture heated 9 hrs., distilling alc. as 400°F. and 37% viscosity were obtained and 27.55% III was added during 1 hr., and esterification continued until the product had 31% viscosity. A fourth resin was prepared in the same way until a viscosity of 37% was reached, V added to yield a solution having 70% solids, and the esterification continued at 425°F. until 34% viscosity was reached. The resins were then applied and cured by standard methods. Sometimes a curing catalyst, e.g., Zn octoate, Cd octoate, Cu naphthenate, or aromatic diisocyanates, was employed when the resin was applied to conductors. The wire enamels were tested by the following tests: Mil cut through temperature; % elongation; 25% elongation + 1X-flexibility; Scott twist; dielec. strength, twisted pair; Emerson scrape; CHCl₃ resistance; and 50:50 solvent resistance. The insulated wires were further improved by applying an overcoat of a highly linear thermoplastic polymer (VII) m. >175°, relative viscosity >1.3, tensile strength 230,000 psi at 175°, and thermal life 24,000 hrs. at 200°. This layer prevents heat shock in the underlying layer and enhances the phys. and chemical properties of the finished wire. Thus, 125 parts Number 54 was dissolved in 875 parts o-cresol and p-chlorophenol (4:1 by weight) at 80-90°, 10 parts xylene added, vacuum applied to remove xylene and water, the mixture heated 30 min. to 110-20°, and the mixture cooled to <60°, resulting in a coatable solution, viscosity 195-200 cps., solids content 12.5%, and weight

8.90 lb./gal. VII can also be blended into the polyesters containing I, and this blend may be cured as a wire enamel.
 ACCESSION NUMBER: 1967:47415 CAPLUS
 DOCUMENT NUMBER: 66:47415
 TITLE: Melamine-aldehyde resin modified polyester reaction products
 INVENTOR(S): George, Norman J.; Kitun, Alexander
 PATENT ASSIGNEE(S): George, P. D., Co.
 SOURCE: U.S., 11 pp.

DOCUMENT TYPE:
LANGUAGE:
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3297785		19670110	US	19630930

L20 ANSWER 156 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

GI For diagram(s), see printed CA Issue.

AB cf. CA 57, 729g. A solution of benzenediazonium chloride prepared from 175 mg.

aniline was added to an ice cold mixture of 250 mg. 3,4-benzotropolone (I) in 25 cc. AcOH and 4.5 g. NaOAc in 13.5 cc. H₂O and the mixture stirred 1 hr. to give 120 mg. 5-phenylazo-3,4-benzotropolone (II), red, m. 148-50° (Me₂CO). When the coupling reaction was carried out in pyridine solution, a dark red tarry product was formed. Chromatography on silica gel in benzene solution gave an unidentified azo compound, m. 171.5-2.5°, in 14% yield. Similarly prepared from 7-bromo-3,4-benzotropolone (III) in AcOH solution was 28% 5-phenylazo-7-bromo-3,4-benzotropolone, brownish orange, m. 168-9°. To an ice cold solution of 500 mg. III in 15 cc. pyridine was added dropwise a solution of benzenediazonium chloride from 240 mg. aniline. The precipitate

formed on dilution with an equal volume of H₂O was filtered off to give 160 mg.

2-phenyl-2H-benz[*g*]indazole-4,5-dione (IV), yellow, m. 251° (EtOH). 2-(*p*-Tolyl)-2H-benz[*g*]indazole-4,5-dione (V), orange yellow, m. 273°, was similarly prepared in 69% yield. The filtrate from IV was concentrated in vacuo to a tarry residue which was dissolved in benzene. Chromatography on silica gel gave 45 mg. 2-phenylazonaphthol (VI), m.p. and mixed m.p. with an authentic sample 134°. The filtrate from V gave no azonaphthol. The coupling of 500 mg. III with 1 equivalent

diazotized *p*-anisidine in pyridine solution gave 65 mg. 2-(*p*-anisylazo)-1-naphthol, red,

m. 124°, and 30 mg. 2,4-bis(*p*-anisylazo)-1-naphthol, dark brown, m. 182.5°. Similar coupling of 7-chloro-3,4-benzotropolone with benzenediazonium chloride in pyridine solution gave a crude product which on chromatography in benzene solution on silica gel gave 85 mg. VI, m.p. and mixed m.p. 134°. IV and V on hydrogenation over Pd-charcoal gave leuco compds. which regenerated the original materials on removal of solvent and catalyst and exposure to air. A suspension of 100 mg. IV and 500 mg. fused NaOAc in 20 cc. Ac₂O was treated with 300 mg. Zn powder. After the mixture became nearly colorless, excess Ac₂O was decomposed with H₂O, the precipitate extracted with hot alc., and the extract concentrated to give 100 mg.

2-phenyl-4,5-diacetoxy-2H-benz[*g*]indazole (VII), m. 191° (EtOH). 2-(*p*-Tolyl)-4,5-diacetoxy-2H-benz[*g*]indazole (VIII), m. 167-8°, was similarly prepared in 77% yield. VII and VIII underwent hydrolysis and autoxidn. to regenerate IV and V when 10-mg. samples were heated with 1.5 cc. EtOH containing 3 drops concentrated HCl. IV with *o*-phenylene-diamine gave IV quinoxaline derivative, m. 233-4° (benzene). V quinoxaline derivative m. 252°. Oxidation of IV or V in pyridine solution with KMnO₄ gave phthalic acid, identified as phthalanil, m.p. and mixed m.p. 205-6°. To a solution of 200 mg. IV in 80 cc. AcOH containing 6 cc. concentrated H₂SO₄ was added

dropwise 5 cc. 30% H₂O₂ during 1 hr. at 40°. After standing 1 hr. at 40°, the solution was concentrated in vacuo to 10 cc. and diluted with 10 cc. H₂O. The precipitated 1-phenyl-3-(*o*-carboxyphenyl)pyrazole-4-carboxylic acid

(IX) m. 284-5° (decomposition) (AcOH), yield 180 mg. Similarly prepared was 1-(*p*-tolyl)-3-(*o*-carboxyphenyl)pyrazole-4-carboxylic acid, m. 300-2°. A mixture of 200 mg. IX and 200 mg. Cu powder was heated at 285-95° for 10 min. at 5 mm. The sublimate gave 130 mg. 1,3-diphenylpyrazole (X), m. 84.0-4.5° (dilute EtOH). M.p., mixed m.p., and ir spectra were identical with those of an authentic sample.

L20 ANSWER 155 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB cf. CA 60, 14104b; 61, 16305h. Kinetics of Me oleate autoxidn. was investigated in the presence of the chelates of diisocylideneethylenediamine with Cu⁺⁺, Fe⁺⁺, Fe³⁺, Zn⁺⁺, Co⁺⁺, Ni⁺⁺. Three g. of substrate (Me oleate 94.3%, Me linoleate and linoleate 5.7%, peroxide number 5) with 1% chelate were incubated in a Warburg apparatus and O consumption measured up to 120 hrs. After oxidation, the substrate was stored under N at -10°, until examined by thin-layer chromatography. The mixture (2% in CHCl₃) was

spotted on silica gel, eluted with benzene-EtOAc (9:1), and developed with 2,7-dichlorofluorescein or H₂SO₄ for total fatty substances, or with KI + starch solution for peroxide-type substances. The substrate was reduced in two ways: by hydrogenation of substrate (200 mg.) in 15 ml. EtOAc with 100 mg. of PtO₂ and separation of the mixture by chromatography, or by reduction of

substrate (200 mg.) in 20 ml. anhydrous ether with LiAlH₄ (60 mg. in 15 ml. anhydrous ether), and separation of obtained alcs. by chromatography. The chelates were used to study the decomposition of cumyl hydroperoxide

(II). 1 (100 mg.) and 10 mg. chelate in 1 ml. *n*-decane were incubated at 40°. Residual peroxide was determined by titration with 0.01N Na₂S₂O₃, after dissolving the incubated mixture in 22 ml. of AcOH-CHCl₃ (3:2), adding 20 ml. of aqueous saturated KI solution, keeping in the dark 1 hr., and adding 75 ml.

of H₂O. A different behavior for chelates was observed and related to their different structure and to activation and type of metal bond. Chelates promoted peroxide formation and their decomposition to non-peroxidic

comps.

ACCESSION NUMBER: 1966:45177 CAPLUS

DOCUMENT NUMBER: 64:45177

ORIGINAL REFERENCE NO.: 64:8509c-f

TITLE: Autooxidation of polyunsaturated fats. V

AUTHOR(S): Fedeli, E.; Valentini, A. F.; Lanzani, A.; Jacini, G.

CORPORATE SOURCE: Staz. Sper. Olii Grassi, Milan

SOURCE: Rivista Italiana delle Sostanze Grasse (1965), 42(10), 488-92

CODEN: RISGAD; ISSN: 0035-6808

DOCUMENT TYPE: Journal

LANGUAGE: Italian

L20 ANSWER 156 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

Coupling of 500 mg. 5-bromo-3,4-benzotropolone with benzenediazonium chloride from 240 mg. aniline gave 150 mg. of an uncharacterized azo compd., Cl⁷H₁₁O₂N₂Br, red, m. 160-1°. Coupling of 500 mg. 5,7-dibromo-3,4-benzotropolone (XI) with benzenediazonium chloride from 185 mg. aniline gave 2-phenylazo-4-bromo-1-naphthol (XII), red, m. 178.5°, yield, prepn. in AcOH soln. 140 mg.; yield in pyridine soln. 110 mg. 2-Phenylazo-4-chloro-1-naphthol (XIII), red, m. 160°, yield 294; and 2-(*p*-tolylazo)-4-bromo-1-naphthol, reddish orange, m. 183-4°, yield 294, were similarly prepd. The latter compd. showed no m.p. depression on mixt. with a sample prepd. by coupling 4-bromo-1-naphthol with *p*-toluene-diazonium chloride in pyridine soln. To a suspension of 100 mg. XII and 500 mg. fused NaOAc in 3 cc. Ac₂O was added 300 mg. Zn powder in portions. Excess Ac₂O was decomposed with H₂O and the ppt. worked up to give 65 mg. 1-acetoxy-2-acetamido-4-bromonaphthalene, m. 232° (EtOAc). XIII similarly gave 68% 1-acetoxy-2-acetamido-4-chloronaphthalene, m. 195-200°. To a soln. of 500 mg. III in a mixt. of 20 cc. 0.4N KOH and 8 cc. pyridine was added dropwise (ice cooling) during 2 hrs. a soln. of 450 mg. K₂S₂O₈ in 15 cc. H₂O. After standing 3 days in an ice chest, 100 cc. concd. HCl was added. The pptd. product, on recrystn. from EtOH gave 210 mg. unchanged III (m.p. and mixed m.p. 147°) and 150 mg. 5(?)-hydroxy-7-bromo-3,4-benzotropolone (XIV), yellow, m. 317-20° after darkening at 180°. A soln. of 300 mg. III in Me ether in a mixt. of 15 cc. MeOH and 1.2 g. 50% KOH was kept for 10 hrs. at room temp. The mixt. was acidified with dil. HCl, concd., and the product worked up to give 165 mg. 6-hydroxy-7-methoxy-2,3-benzotropolone-H₂O, m. 103.5-4.5° (dil. EtOH). The anhyd. product (XV) was obtained by drying over P₂O₅ 2 days. A soln. of 100 mg. XV in 2 cc. concd. HCl was heated in a sealed tube at 135-40° 5 hrs. The soln. was dild. to give 65 mg. 6,7-dihydroxy-2,3-benzotropolone, m. 133.5° (H₂O). Bromination of 200 mg. XV in 2 cc. AcOH with 320 mg. Br gave an immediate pptn. of a reddish violet intermediate product. The whole was heated 1 hr. on a water bath and the mixt. dild. to give 4(?)-bromo-6,7-dihydroxy-2,3-benzotropolone, m. 198.5° (EtOH). The intermediate product regenerated XV on heating in H₂O. After standing overnight at room temp., a soln. of 300 mg. XI in Me ether in 60 cc. MeOH contg. 1.2 g. 50% KOH was acidified with dil. HCl and evapd. to give 180 mg. Me 1-hydroxy-4-bromo-2-naphthoate, m. 120-1° (MeOH). Reaction with diazomethane gave Me 1-methoxy-4-bromo-2-naphthoate, m. 94°. Ir data were given.

ACCESSION NUMBER: 1966:43609 CAPLUS

DOCUMENT NUMBER: 64:43609

ORIGINAL REFERENCE NO.: 64:8105d-h, 8106a-f

TITLE: 3,4-Benzotropolone and related compounds. IV. Azo- and hydroxy-3,4-benzotropolones

AUTHOR(S): Ebine, Seiji

CORPORATE SOURCE: Saitama Univ., Urawa

SOURCE: Bulletin of the Chemical Society of Japan (1965), 38(12), 2029-34

CODEN: BCSJAB; ISSN: 0009-2673

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 64:43609

L20 ANSWER 157 of 253 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA issue.
AB The title compds. (Ic-g) show antiinflammatory, analgesic, antipyretic, and antiradical activity; they are prepared by treating Ib with 5 and a secondary amine, and hydrolysis of the corresponding thioamide to Ic (A = CH₂). I (R = COCl) can be treated with CH₂N₂, followed by the reaction with Ag₂O and MeOH to give Ic (A = CH₂, R = Me), eventually followed by hydrolysis to the free acids Ic (A = CH₂, R = alkyl) can be treated with (EtO)₂CO and MeONa to give Id, which is treated with an alkyl halide to give Ic (A = α-branched alkyl); Ia can be treated in the presence of AlCl₃ with a haloacyl ester to give Ic (A = CO(CH₂)_n); Id can be made to react with CH₂O and NMe₂, followed by hydrolysis, to give Ic (A = CH(CH₂)). Thus, a dispersion of 14.5 g. AlCl₃ in 60 ml. PhNO₂ is treated at 10° with 9 ml. AcCl and 17.69 g. Ia (R₁ = H, R₂ = 4-Cl), under stirring. After 5 hrs., the mixture is decomposed on ice and HCl. The solvent is steam-distilled and the crystallization residue worked up to give Ib (R₁ = H, R₂ = 4-Cl), m. 96-103° (EtOH), of which 17.87 g. is refluxed 7 hrs. with 4 g. 5 and 23 ml. morpholine. EtOH (25 ml.) is added, and the precipitate is separated and washed with 50 ml. EtOH. The thiomorpholide is refluxed 8 hrs. with 140 ml. 70% EtOH and 50% NaOH. The alc. is evaporated, the residue is diluted with hot H₂O and worked up to give Ib (R₁ = H, R₂ = 4-Cl, A = CH₂, R₃ = H), m. 158-61° (EtOAc). A solution of 180 g. of the acid in 500 ml. EtOH and 50 ml. concentrated HCl is refluxed overnight. The alc. is distilled and the residue diluted with H₂O and worked up to give Ic (R₁ = H, R₂ = 4-Cl, A = CH₂, R₃ = Et), m. 65° (petroleum ether, b. 40-60°). With stirring, NaOEt (from 12.9 g. Na and 325 ml. EtOH) is added to a solution of 123 g. of the ester in 710 ml. (EtO)₂CO. The solution is distilled till 124° is obtained at the head of the distillation column, and the residue is cooled overnight at 0°, then treated with 62 ml. AcOH in 250 ml. H₂O and extracted with Et₂O. Working up of the extract gives Id (R₁ = H, R₂ = 4-Cl, R₃ = Et), m. 47-9° (petroleum ether, b. 40-60°). The ester (34.7 g.) is added with stirring to a solution of NaOEt (from 2.5 g. Na and 100 ml. EtOH), and 16.5 ml. MeI is added. The solution is refluxed 2 hrs., the alc. is evaporated, and the residue is hydrolyzed by refluxing 5 hrs. with 200 ml. 2.5N NaOH and 100 ml. EtOH. The precipitate is separated and heated 1 hr. at 190° to give Ic (R₁ = H, R₂ = 4-Cl, A = CH₂, R₃ = H), m. 175-5° (EtOH). A solution of 136 ml. Ac₂O in 1560 ml. CS₂ is added with stirring to a mixture of 302.5 g. Ia (R₁ = H, R₂ = 2-Cl) and 480 g. AlCl₃ in 1000 ml. CS₂. The mixture is refluxed 2 hrs., the solvent evaporated, and the residue poured on ice-HCl. The solution is extracted with Et₂O and working up gives Ib (R₁ = H, R₂ = 2-Cl), m. 64-6° (petroleum ether, b. 62-8°). A solution of 240 g. 3-CH₂CH₂CH₂CH₂ in 280 ml. HCl and 160 ml. H₂O is diazotized with 130 ml. NaNO₂ in 150 ml. H₂O. The diazonium salt is separated and stirred with 1.8 l. C₆H₆ at 0-10°, 330 g. NaOAc, 3H₂O in 500 ml. H₂O added, and the stirring is continued 48 hrs. Working up of the benzene layer gives Ia (R₁ = H, R₂ = 3-NO₂) as an oil, to which (194 g.), in a mixture with 400 g. AlCl₃ and 2.5 l. CS₂, is added dropwise 152 ml. AcCl. The solution is refluxed gently 4 hrs., poured on ice, and concentrated. Working up of the organic phase gives Ib (R₁ = H, R₂ = 3-NO₂), m. 110-11.5° (EtOH), of which 98 g. is added portionwise, with stirring to a solution of 280 g. SnCl₂.2H₂O

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114-15°. A mixture of 5 g. of this nitrate, 25 ml. AcOH, and 50 ml. HCl is refluxed 18 hrs., then dild. with H₂O and the ppt. is sepd. Working up of the ppt. gives Ic (R₁ = H, R₂ = 2-NO₂, A = CH₂, R₃ = H), m. 142-5° (C₆H₆, petroleum ether). A soln. of 35 g. of nitrile in 350 ml. EtOH and 5.4 ml. H₂O is satd. at ambient temp. with gaseous HCl. The soln. is refluxed 3 hrs., poured on aq. NaHCO₃ and extd. with a mixt. of EtOAc and Et₂O. Working up of the ext. gives Ic (R₁ = H, R₂ = 2-NO₂, A = CH₂, R₃ = Et), b. 15-196-202° m. 73-5°. A soln. of 19 g. of the ester in 300 ml. EtOH is hydrogenated, using 2 g. of a Pd-C catalyst. When 5.1 l. H₂ is absorbed, the catalyst is filtered, and working up gives Ic (R₁ = H, R₂ = 2-NH₂, A = CH₂, R₃ = Et), b. 15-198-200°. A soln. of 13.6 g. of this ester in 250 ml. 5N H₂SO₄ is diazotized with 4.1 g. NaNO₂. The filtered diazonium soln. is added to boiling 6N H₂SO₄; the soln. is refluxed 15 min. and poured on ice. Extn. with Et₂O and working up of the ext. gives Ic (R₁ = H, R₂ = 2-OH, A = CH₂, R₃ = H), m. 152-4° (C₆H₆). A mixt. of 0.5 g. Ic (R₁ = 2-OH, R₂ = H, A = CH₂, R₃ = H), 3 ml. MeI, and 10 ml. Me₂CO is refluxed 17 hrs. with 0.9 g. K₂CO₃. The Me₂CO is distd., 10 ml. 2N NaOH, and 5 ml. EtOH are added, and the soln. is refluxed 1 hr. The EtOH is distd. and the residue is acidulated with dil. HCl. Working up of the ppt. gives Ic (R₁ = 2-MeO, R₂ = H, A = CH₂, R₃ = H), m. 108-11° (petroleum ether). A soln. of 7 g. 1 (R₁ = 2-NO₂, R₂ = CO₂H, R₃ = H) and 70 ml. SOCl₂ is refluxed 45 min. The excess SOCl₂ is evapd. in vacuo and two 10-ml. portions dry C₆H₆ are added and distd. in vacuo. The residual acyl chloride is dissolved in 70 ml. dry Et₂O, and added at -15° to -20° to CH₂N₂ soln. (obtained from 30 g. MeNHCONH₂ and 100 ml. Et₂O). After 1 hr. stirring, the suspension is left overnight at ambient temp., and the ppt. is worked up to give I (R₁ = 2-NO₂, R₂ = H, R₃ = COCH₂NH₂), m. 128-33° (decompn.) (MeOH). To a soln. of 0.3 g. of the diazo ketone in hot MeOH, 0.5 g. freshly prepd. Ag₂O is added portionwise at 50-5°. After 3 hrs. at this temp., the mixt. is filtered, and the filtrate evapd. to dryness. The residue is taken up in Et₂O, the soln. filtered and evapd. Working up of the residue gives Ic (R₁ = 2-NO₂, R₂ = H, A = CH₂, R₃ = Me), m. 68-70° (petroleum ether, b. 62-8°). Hydrolysis with MeOH and 0.1N NaOH gives Ic (R₁ = 2-NO₂, R₂ = H, A = CH₂, R₃ = H), m. 154-6.5° (C₆H₆). A mixt. of 10 g. Ic (R₁ = H, R₂ = 2-F, A = CH₂, R₃ = H), 2.5 ml. concd. H₂SO₄, and 75 ml. EtOH is refluxed 5 hrs. The excess alc. is evapd. and the residue dild. with H₂O. The ester is extd. with Et₂O and working up gives Ic (R₁ = H, R₂ = 2-F, A = CH₂, R₃ = Et), m. 42-8°, b. 163°. A mixt. of 3.3 g. Ic (R₁ = H, R₂ = 2-Cl, A = CH₂, R₃ = H) and 6.6 ml. SOCl₂ is refluxed 1 hr. and the excess SOCl₂ is evapd. The residue is poured in 20 ml. ice-cool. NH₄OH (d. 0.88), and the ppt. is sepd. to give Ia (R₁ = H, R₂ = 2-Cl, A = CH₂, R₃ = H), m. 139-41° (EtOH). Ib (R₁ = 2-NH₂, R₂ = H) is diazotized and the diazonium salt is added to K methyloxanthate. The soln. is extd. with Et₂O and working up gives Ib (R₁ = 2-SMe, R₂ = H), m. 90-2°. Ic (R₁ = 2-Br, R₂ = H, A = CH₂, R₃ = Me) (2.57 g.), 0.95 g. CuCN, and 4 ml. HCO₂Me₂ is refluxed 4 hrs., and the soln. is poured in a warm soln. of 3.5 g. FeCl₃, 4 ml. concd. HCl, and 20 ml. H₂O. The mixt. is left overnight and the ppt. sepd. and worked up to give Ic (R₁ = 2-Cl, R₂ = H, A = CH₂, R₃ = Me), m. 87-5-9.5° (petroleum ether), and alk. hydrolysis gives Ic (R₁ = 2-Cl, R₂ = H, A = CH₂, R₃ = H), m. 152-5.5° (C₆H₆). Type, R₂, R₁, A, R₃, NB, b.p. or m.p.: Ib, 2-F, H, --, --, 84-5.5°; Ib, 4-Cl-2-Me, H, --, --, 80-2°; Ib, 3-F, H, --, --, 91-3°; Ic, 2-Cl, H, CH₂, Et, --b. 15-172-4°; Ic, 2-Cl, H, CH₂, H, --, 116-5-19.5°; Ic, 3-Cl, H, CH₂, H, --, 129-31°; Ic, 2,6-F₂, H, CH₂, H, --, 132-3°; Ic, 2-F, CH₂, H, --, 143-4.5°; Ic, H, 2-Cl, CH₂, H, --, 106-7°; Ic, H, 2-Br, CH₂, H, --,

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in 1.7 l. concd. HCl and 500 ml. EtOH. The soln. is heated 4 hrs. on a steam bath, then cooled and poured on a mixt. of ice and 40% NaOH. The ppt. is sepd. and worked up to give Ib (R₁ = H, R₂ = 3-NH₂), m. 159-62° (EtOH), of which a soln. of 21.1 g. in 125 ml. 4N HCl is diazotized with 7.6 g. NaNO₂ in 25 ml. H₂O. The diazonium soln. is filtered and added dropwise, with vigorous stirring, to a soln. of CuCl in HCl. The soln. is heated 1.5 hrs. on a steam bath, then poured on ice, extd. with Et₂O and the ext. worked up to give Ib (R₁ = H, R₂ = 3-Cl), oil. A soln. of 140 g. 2,5-H₂NCH₂CH₂Me in 2.5 l. C₆H₆ is refluxed 18 hrs. with a soln. of 175 ml. amyl nitrite in 500 ml. C₆H₆. The solvent is distd. and fractionation of the residue gives Ia (R₁ = H, R₂ = 4-Cl, 2-Me), b. 80-8-109-11°. A soln. of 54 g. AlCl₃ in 140 ml. PhNO₂ is added dropwise, with stirring, to a soln. of 30 g. Ia (R₁ = R₂ = H), and 38.5 g. EtOOC(CH₂)₄COCl 100% PhNO₂, keeping the temp. between 5-10°. The mixt. is stirred 4 hrs., left overnight, and decomposed on ice-HCl. The ppt. is sepd., the solvent from the filtrate is steam-distd., and the residue, together with the initial ppt., is worked up to give Ic (R₁ = R₂ = H, A = CO(CH₂)₄, R₃ = Et), m. 96-8°. A soln. of 3.8 g. of the ester in 50 ml. EtOH and 10 ml. 5N NaOH is refluxed 2 hrs., the alc. is evapd. in vacuo, the ppt. dissolved in hot aq. NaHCO₃, and workup gives Ic (R₁ = R₂ = H, A = CO(CH₂)₄, R₃ = H), m. 159-62.5° (EtOH). A soln. of 40 g. of the acid in 75 ml. H₂O, 175 ml. concd. HCl, and AcOH is treated with a 2n-analys. (prepd. from 100 g. 2n and 10 g. HgCl₂, 5 ml. concd. HCl, and 150 ml. H₂O), and refluxed 28 hrs.; 50 ml. HCl is added after 2, 4, and 6 hrs. The mixt. is cooled, dild. with H₂O and the sepd. oil is taken up in Et₂O. Working up gives Ic (R₁ = R₂ = H, A = CH₂5, R₃ = H), m. 103-5° (petroleum ether, b. 80-100°). A suspension of 68 g. Id (R₁ = R₂ = R₃ = H) in 100 ml. H₂O is cooled, and 96 ml. of an aq. 2.84N soln. of NMe₂ is added. The soln. is cooled in ice and 32 ml. of a 38% CH₂O soln. is added dropwise. The stirring is continued overnight at ambient temp., the soln. is filtered, and HCl is added to the filtrate till pH = 4. The ppt. is sepd., suspended in 150 ml. H₂O, neutralized (NaOH) and refluxed overnight in a N atm. The mixt. is cooled, acidulated and the ppt. is worked up to give Ic (R₁ = R₂ = H, A = CH(CH₂)₂, R₃ = H), m. 180-2° (EtOH, H₂O). A soln. of 258 g. 4,2-BrMeC₆H₃NH₂ in 1.5 l. C₆H₆ is added to 240 ml. butyl nitrite in 1.5 l. C₆H₆; the soln. is left overnight at ambient temp. and refluxed 12 hrs. The solvent is distd. and the residue extd. with light petroleum ether. Working up of the ext. gives I (R = Br, R₁ = 2-Me, R₂ = H), b. 7-114°, of which 61.7 g., together with 26.8 g. CuCN, is added to 40 ml. HCO₂Me₂, is refluxed 4 hrs. The hot soln. is poured on a mixt. of 105 g. FeCl₃, 25 ml. concd. HCl, and 150 ml. H₂O, and this soln. is heated 20 min. at 60-70°. The hot soln. is extd. 6 times with 200 ml. PhMe. Working up of the ext. gives I (R = CN, R₁ = 2-Me, R₂ = H), m. 37-8°. A soln. of 10.2 g. of the nitrile in 70 ml. dry Et₂O is added dropwise at ambient temp., to a soln. of MgMeI (from 6.5 g. Mg and 36.3 g. MeI) in 80 ml. Et₂O. The soln. is refluxed 5 hrs. with stirring, and stirring is continued overnight at ambient temp. The complex is decomposed with 200 ml. 2N HCl, while distg., the Et₂O, and the hydrolysis is completed by heating 15 min. at 90-100°. Extn. with 1:1 Et₂O-Me₂CO and working up of the residual red resin gives Ib (R₁ = 2-Me, R₂ = H), m. 84-5°. Dry HCl gas is bubbled 18 hrs. through a soln. of 22 g. Ia (R₁ = H, R₂ = 2-NO₂), 150 g. anhyd. 2NCl₂, and 100 g. (CH₂O)₃ in 400 ml. glacial AcOH. The mixt. is poured on ice, extd. with Et₂O and the ext. worked up to give I (R = CH₂Cl, R₁ = H, R₂ = 2-NO₂), m. 89-90°, of which 49.5 g. in 125 ml. Me₂SO is added dropwise, with stirring, to a suspension of 15 g. NaCN in 150 ml. Me₂SO. The mixt. is kept 2 hrs. at 40-60° then poured on ice, and extd. with Et₂O. Working up of the ext. gives I (R₁ = H, R₂ = 2-NO₂, R = CH₂CN), m.

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114-16°. Ic, H, 3-OMe, CH₂, H, --, 162-4°. Ic, 2-F, H, CH₂, H, --, 101-3° (1); Ic, 2-F, H, CH₂, CH₂CH₂NH₂, --, b. 170-2° (2); Ic, 2-Me, H, CH₂, H, --, 108-10°. Ic, 2-Et, H, CH₂, H, --, 82-3°. Ic, 2,3-Me₂, H, CH₂, H, --, 172-4°. Ic, H, 2-Me, CH₂, H, --, 107-8°. Ic, H, 2-Me, CH₂, Et, --, b. 6 152-3°. Ic, H, 3-F, CH₂, H, --, 178-80°. Ic, H, 3-Cl, CH₂, H, --, 142-5.5°. Ic, H, 2-Et, CH₂, H, --, 69-70°. Ic, H, 2-CF₃, CH₂, H, --, 101-3°. Ic, H, 2,3-Me₂, CH₂, H, --, 143-5°. Ic, 3-F, CH₂, H, --, 144°. Ic, 2-MeO, H, CH₂, H, --, 168-70.5°. Ic, H, 2-OBt, CH₂, H, --, 70.5-2°. Ic, H, 2-CH₂CH₂CH₂CH₂, CH₂, H, --, 67.5-70°. Ic, H, 2-SMe, CH₂, H, --, 97.5-99°. Ic, 2-F, 2-F, CH₂, H, --, 126-7°. Ic, 4-Cl, H, CH₂, H, --, 153-4°. Ic, H, 2-Br, CH₂, H, --, 133-5°. Ic, H, 2-F, CH₂, H, --, 110-11°. Ic, 2-Cl, H, CH₂, H, --, 110-12°. Ic, 2-Cl, H, CH₂, Et, --, b. 14 166-8°. Ic, 2-F, H, CH₂, H, --, 94.4-7°. Ic, H, 2-Me, CH₂, H, --, 101-3°. Ic, H, 2-Me, CH₂, H, --, 111-13.5°. Ic, H, H, CO(CH₂)₃, Et, --, 89.5-92°. Ic, H, H, CH₂9, H, --, 112-14.5°. Id, 2-Cl, H, --, Et, --, b. 0.075 172-8°. Ic, 2-F, H, CH₂, --, NH₂ 149-50°. Ic, 2-F, H, CH₂, --, NH₂, b. 15 172-4°. If, 2-Cl, H, CH₂, --, --, b. 0.2 143-54°. (1) Pyridine salt m. 138-40°, NH₄ salt m. 132-5°. (2) HCl salt m. 125°. Ic (R₁ = H, R₂ = 2-Cl, A = CH₂, R₃ = Et) (7 g.) is added slowly with stirring to a soln. of 1.26 g. LiAlH₄ in 53 ml. Et₂O. The mixt. is refluxed 1 hr. and decomposed with H₂O, and the org. layer sepd., extd. with dil. HCl, the aq. ext. made alk. (NaOH) and extd. with Et₂O. Working up of the Et₂O ext. gives Ig (R₁ = H, R₂ = 2-F, A = CH₂, NB = NEt₂) 2, b. 2 126-30°; EtCl salt m. 134-6° (EtOH, Et₂O). Id (R₁ = H, R₂ = 2-Cl, R₃ = Et) (18 g.) is added to a soln. of 0.57 g. Na in 30 ml. EtOH and 4 ml. allyl bromide is added. The mixt. is refluxed 2 hrs., and the excess alc. is evapd. in vacuo, 36 ml. 2.5N NaOH and 18 ml. EtOH are added and the mixt. is heated 2 hrs. on a steam bath. The alc. is evapd. and the pptd. Na salt is dissolved in hot H₂O, the soln. is acidulated (HCl), and the substituted malonic acid is extd. with Et₂O. The Et₂O is evapd. and the residual oil is heated 20 min. at 160-75° to give Ic (R₂ = 2-Cl, R₁ = CH(CH₂CH₂CH₂), R₃ = H), m. 74-6° (petroleum ether, b. 40-60°). A soln. of 58 g. 2-BrC₆H₄NO₂ and 81 g. 2,5-Br₂C₆H₃NO₂ in 250 ml. PhNO₂ is heated 15 min. at 170-80°, with addition of 70 g. Cu powder. The mixt. is stirred 15 min. at 180°, cooled, and filtered. The filtrate is vacuum distd. at 100° and the residue is rubbed with MeOH to give a solid. Working up gives I (R₁ = R₂ = 2-NO₂, R = Br), m. 120-4° (MeOH). This dinitro deriv. (50 g.) in a Soxhlet, is extd. 30 min. at reflux, with a boiling mixt. of 750 ml. EtOH, 330 g. SnCl₂.2H₂O, and concd. HCl. The solvent is evapd., aq. NaOH is added to dissolve the inorg. ppt., and the soln. is extd. with Et₂O. Working up of the ext. gives I (R₁ = R₂ = 2-NH₂, R = Br), m. 61-2° (petroleum ether, b. 62-8°). To a soln. of 10 g. of the diamine in 20 ml. tetrahydrofuran and 38 ml. 40% fluoroboric acid at 0°, 6 g. PhNO₂ in 10 ml. H₂O is added. The mixt. is stirred 15 min. at 0° and the red-brown ppt. is sepd., washed with 4% fluoroboric acid, an Et₂O-MeOH (9:1) mixt., and Et₂O. The dry fluoroborate is suspended in 100 ml. dry xylene and refluxed with stirring while BF₃ escapes. The mixt. is heated 1 more hr., cooled, and stirred 30 min. with 100 ml. 5N NaOH. Working up of the org. layer gives I (R₁ = R₂ = 2-F, R = Br, A = 45-6°

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(petroleum ether b. 40-60°). The tabulated I were prep'd. using these methods.

ACCESSION NUMBER: 1966:27315 CAPLUS
DOCUMENT NUMBER: 64:27315
ORIGINAL REFERENCE NO.: 64:5005e-h, 5006a-b, 5007a-h, 5008a-e
TITLE: Preparation of substituted biphenyl derivatives
PATENT ASSIGNEE(S): Boots Pure Drug Co., Ltd.
SOURCE: 34 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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(NO3)2, o-phenanthroline, and Et3N in MeOH (or EtOH) at 20°, gave 30-50% AcCH:CHCMe2OH (XI), b1 80-5°, n20D 1.4740, 10% β,β-dimethylglycidialdehyde (XII), and 10-35% Me (or Et) β,β-dimethylglycidate, b15 61-5°, n20D 1.4240.

Hydrogenation of XI over Pd/C in AcOEt gave a mixt. of IX and X. XI (6.6 g.) and 200 ml. 1% H2SO4 kept 24 hrs. at 65° gave 81% AcCH:CHCMe2CH2, b15 67-9°, n20D 1.4965. Oxidn. of IX was assumed to proceed via the γ hydroperoxide, AcCH:CHCMe2OOH (XII). XII, m. 29-30° (pentane-ether), was prep'd. by oxidn. of an equil. mixt. of IX and X at 59° with O and with Me2C(CN)N:NCMe2CN as initiator. Decompn. of XII under the conditions used for oxidn. of IX gave the same products as IX and in about the same yields. The mechanism is thought to involve intramol. rearrangement of an alkoxy radical formed from XII, leading to a radical contg. an epoxide ring. The latter is converted by O to a peroxy radical, which is converted to 4,5-epoxy-2,3-hexanedione and thence to the observed products. Photochem. oxidn. of EtCH:CHMe2 with O in MeOH in the presence of Rose bengal as a sensitizer gave a mixt. of hydroperoxides. This mixt. with O in MeOH in the presence of Cu-(NO3)2, o-phenanthroline, and MeONa gave 12% 3,4-epoxy-4-methyl-2-pentanone and 63% Me2C(OH)CH:CHMe. Infrared, N.M.R., and mass spectra were in agreement with the proposed structures.

ACCESSION NUMBER: 1966:27036 CAPLUS
DOCUMENT NUMBER: 64:27036
ORIGINAL REFERENCE NO.: 64:4932c-h, 4933a-c
TITLE: The copper-catalyzed oxidation of unsaturated carbonyl compounds. III. Oxidation of α,β- and β,γ-unsaturated aldehydes and ketones by oxygen in the presence of cupric complexes
AUTHOR(S): Volger, H. C.; Brackman, W.; Lemmers, J. W. F. M.
CORPORATE SOURCE: Koninkl. Shell Lab., Amsterdam
SOURCE: Recueil des Travaux Chimiques des Pays-Bas (1965), 84(9-10), 1203-29
CODEN: RCTPAA; ISSN: 0165-0513
DOCUMENT TYPE: Journal
LANGUAGE: English

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AB cf. CA 63, 13015h. Certain α,β- and β,γ-unsatd. carbonyl compds. may be oxidized under mild conditions by O in the presence of a base and catalytic amts. of Cu(II) complexes in MeOH solution. The reaction is restricted to compds. which may be converted by deprotonation to dienolate anions containing the skeleton C=C-C-C-O-.

The oxidation takes place at the γ-position, giving a tertiary alc., ketone, or aldehyde, depending on the number of H atoms available at the γ-C. The alcs. can be isolated as such, whereas products containing carbonyl functions may form cyclization products such as unsatd. lactones, polyacetals, or cyclic ethers, or may be cleaved into esters or aldehydes. The mechanism is thought to involve oxidation of the dienolate anion by Cu(II) to a dienolate radical, which is converted to a hydroperoxide anion by addition of O to the γ-C and reduction by Cu(I). When 124 millimoles MeCH:CHCHO in 20 ml. MeOH was added with stirring to 4 millimoles Cu(OAc)2, 63 millimoles pyridine, and 36 millimoles Et3N in 60 ml. MeOH under O, there was a rapid uptake of O totaling 102 millimoles to yield 50-70% OHCH(OMe)CH2CHO (I), b19 80-5°, n20D 1.4406, d20 1.114, and 18% MeOCHMeCH2CHO, b75 63-5°, n20D 1.4095 (2,4-dinitrophenylhydrazones m. 106°). On standing I gave a glass-like polymer, presumably formed from the cyclic hemiacetal. Both I and the polymer afforded butenedial bis(2,4-dinitrophenylhydrazones), m. 270-80°, and butenedial bis-(phenylhydrazones), m. 169-70°. I refluxed in dry MeOH containing p-MeC6H4SO3H gave 62% 2,3,5-trimethoxytetrahydrofuran, b38 74-5°, along with (MeO)2CHCH:CHCH(OMe)2, b15 85-7°, n20D 1.4310, and (MeO)2CHCH(OMe)CH2CH(OMe)2, b15 94-7°, n20D 1.4208. BzCH:CHMePh (10 ml.) added to a vigorously stirred solution of 4 millimoles Cu(OAc)2, 5 ml. pyridine, and 5 ml. Et3N in 80 ml. MeOH at 16° gave after 24 hrs. 42% 4,4'-bis(2,4-diphenyl-2-buten-4-olide) (II), m. 275-80° (decomposition) (PhNO2), 8% 2,4-diphenylfuran, and 36% BzOH. II was thought to be formed via BzCH:CPHCHO (III) and the lactone, 2,4-diphenyl-2-buten-4-olide (IV). That III is the precursor of II was supported by the fact that BzCH:CPHCH(OMe)2 (V) gave II in 15% yield on acidic hydrolysis followed by oxidation with Cu(OAc)2, O, pyridine, and Et3N in MeOH. V, b15 120-5°, was prepared in 15% yield from 24 g. BzMe, 54 g. BzCH(OMe)2, and 50 ml. MeOH stirred 4 hrs. with 10% aqueous NaOH. IV, m. 108-10° (EtOH), was prepared in 73% yield from BzCH2CHPhCO2H. IV was rapidly oxidized in alkaline medium giving 50% II and 43% BzCH:CPHCO2H, m. 124-6° (CHCl4). I refluxed 4 hrs. with Zn-Hg in HOAc under N gave 83% IV. I refluxed 16 hrs. with MeONa in MeOH gave 45% BzCH2CHPhCO2Me, m. 107-7.5°, and 25% BzCH:CPHCO2Me. Oxidation of AcCH2CMe:CH2 (VI) with Cu(OAc)2, O, pyridine, and Et3N in MeOH at 0° gave, after concentration of the product by distillation at reduced pressure, 10-35% 5-methoxy-2,4-dimethylfuran, b12 37-8°, n20D 1.4567, and 10% 2,4-dimethyl-2-buten-4-olide (VII), along with some alkaline cleavage products of the intermediate dione (CH2:CHCCHO, HOAc, and HCO2Me). When the reaction mixture in oxidation of VI was neutralized or slightly acidified before concentration, the products included 64% VII, b0.03 48-60°, n20D 1.4523, and 10% 4,4'-bis(2,4-dimethyl-2-buten-4-olide) (VIII) which appeared to be a mixture of 2 isomers. VII, like IV, was rapidly oxidized in alkaline solution by O, even in the absence of the Cu(II)-pyridine catalyst, giving 49% VIII, m. 163-6°. Catalytic oxidation of AcCH:CHMe2 was negligibly slow compared to that of VI. Oxidation of an equilibrium mixture of AcCH2CMe:CH2 (IX) and AcCH:CHCMe2 (X) (containing 75% IX) by O in the presence of Cu

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AB The hydrocarbons which were generated in the catalytic reduction of fatty esters to obtain higher alcohols under high pressure H were investigated. Methyl palmitate, cetyl alcohol, and cetyl acetate were treated under high pressure H (initial pressure 100. approx. 200 atmospheric) in the presence of various kinds of catalysts, such as Cu-Cr-O, Zn-Al-O, Fe-Al-O, Fe-Cu-Al-O, Fe2O3, reduced Fe, ZnO, Al2O3, CuO, Cr2O3, and Raney Ni (W-7). The amounts and the constituents of the generated hydrocarbon depended on catalyst used and reaction temperature. In the case of reduced Fe and metal oxides, the generation of a hydrocarbon having 16 carbons, considered to be produced by dehydration of the alcohol, was preferred to the generations of a hydrocarbon having 15 carbons, considered to be produced by elimination of a methylol group from alcohol. The order of its priority was as follows, Fe-Cu-Al-O > ZnO, CuO, reduced Fe, Al2O3 > Zn-Cr-O, Fe-Al-O > Cr2O3, Fe2O3 > Cu-Cr-O. In the case of the Cu-Cr-O catalyst, there was no appreciable difference in the amount of both hydrocarbons generated. On the other hand, in the case of Raney Ni the generation of C15 hydrocarbon was predominant, and the generation of C16 hydrocarbon was almost the same as those of C14 and less carbon number hydrocarbons. In spite of the presence of high pressure H, the content of olefin in hydrocarbon formed increased in the order Fe-Al-O < Zn-Al-O < Zn-Cr-O < Cr2O3 < ZnO < Al2O3 < Fe2O3. At the same reaction temperature, about 300°, the ease of generation of hydrocarbon was as follows: Raney Ni > Cu-Cr-O > Fe-Cu-Al-O > reduced Fe > Fe-Al-O > Zn-Al-O, ZnO > Cr2O3 > CuO > Fe2O3, Zn-Cr-O, Al2O3. The optimum reaction temperature to produce higher alcohol was different for each of the catalysts and the amount of hydrocarbon formed increased in the order Fe-Al-O > Zn-Al-O > Zn-Cr-O > Cu-Cr-O.

ACCESSION NUMBER: 1965:497610 CAPLUS
DOCUMENT NUMBER: 63:97610
ORIGINAL REFERENCE NO.: 63:17869b-d
TITLE: On the hydrocarbons generated in catalytic reduction of fatty esters
AUTHOR(S): Ikeda, Isao; Hattori, Shozo; Komori, Saburo
CORPORATE SOURCE: Univ. Osaka, Japan
SOURCE: Yukagaku (1965), 14(9), 510-16
CODEN: YKGGAM; ISSN: 0513-398X
DOCUMENT TYPE: Journal
LANGUAGE: Japanese

L20 ANSWER 160 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Passing vaporized MeOH and steam over a supported Ni catalyst eliminates some impurities, which would otherwise poison oxide catalyst used for cracking MeOH to produce H for fuel cell or other applications. Thus, a H₂O-MeOH mixture was passed first through a supported Ni formate catalyst, and then through a pelleted catalyst consisting of Zn, Cr, and Cu oxides in a ratio of approx. 2 parts Zn, 1 part Cr, and 0.14 part Cu. Conditions were: atmospheric pressure, mol. ratio of H₂O to MeOH, 2:1, flow rate, 0.5 volume MeOH/volume oxide catalyst/hr., temperature sufficiently high to maintain 90% conversion of MeOH. Because of catalyst poisoning it was necessary to increase the temperature by 60° (to 350°) in a similar run not using Ni catalyst, and in another case by 40° (to 315°) to maintain 90% conversion. In 2 runs with Ni catalyst, it was only necessary to increase temperature by 35° (to 305°) and by 10° (to 270°).

ACCESSION NUMBER: 1965:486468 CAPLUS
 DOCUMENT NUMBER: 63:86468
 ORIGINAL REFERENCE NO.: 63:15890a-c
 TITLE: Catalytic cracking of methanol
 INVENTOR(S): Holmes, Peter D.; Thornhill, Alan R.
 PATENT ASSIGNEE(S): British Petroleum Co. Ltd.
 SOURCE: 15 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
BE 646977		19641023	BE	
GB 1010574			GB	
PRIORITY APPLN. INFO.:			GB	19630423

L20 ANSWER 162 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB It was proposed to extract Co from HCl solns. with tertiary amine hydrochlorides, R₃N.HCl, as (R₃NH₂)₂CoCl₄, sep. the organic phase, and reext. Co++ with H₂O. Replacing HCl solns. with neutral CaCl₂ solns. as a source of Cl- increased Co extraction from a maximum 75% at 300 g. Cl-/l. to 100% at 375 g. Cl-/l. The extractant was 0.1M C7-9 tertiary amine hydrochlorides in kerosene containing 5% C7-9 alcs. to prevent the formation of a 3rd phase. Increasing Cl- concentration 0-400 g./l. increased the extraction of Co, Cu, Fe(II), and As(III). Zn and Fe(III) were extracted completely at Cl- <100 g./l. The elements Ni, As(V), Mn, and Pb were little extracted. In a proposed flow sheet, Fe+++ is extracted with amine from an acidic solution, Cu is precipitated with H₂S on Co shavings, CaCl₂ is added to a Cl-concn. of 300 g./l., and Co++ is extracted with amine. The extract is treated with H₂O or recycled CoCl₂ solution to recover a purified CoCl₂ solution

ACCESSION NUMBER: 1965:72345 CAPLUS
 DOCUMENT NUMBER: 62:72345
 ORIGINAL REFERENCE NO.: 62:12798e-f
 TITLE: Separation of cobalt from metallurgical solutions by extraction with tertiary amines
 AUTHOR(S): Ioffe, E. Sh.; Dushkina, L. V.
 SOURCE: Tsvetnyye Metally (Moscow, Russian Federation) (1965), 38(2), 36-40
 CODEN: TVMTAX; ISSN: 0372-2929
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 161 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The exchange reaction of O of lactose with that of H₂O does not occur in 10 hrs. at 100°. When a very small amount of a Sn(II), Pd, or Cd salt is added, the reaction occurs, the order of catalytic activity being Pd > Cd > Sn. AgNO₃ has no activity. This order coincides with that of the ionization potentials of these metals. During this reaction, the OH group of lactose is bound to a metallic ion, and the C-OH bond is then loosened by electron displacement. The order of activities of other ions, Cu++ > Zn++ > Ni++ > Fe++ > Mn++, also coincides with the order of complex-forming abilities of these ions.

ACCESSION NUMBER: 1965:467448 CAPLUS
 DOCUMENT NUMBER: 63:67448
 ORIGINAL REFERENCE NO.: 63:12401d-f
 TITLE: Exchange reaction of oxygen atoms between lactose and water in the presence of certain metallic ions as catalysts
 AUTHOR(S): Goto, Kokichi; Matsuoka, Nobuko
 CORPORATE SOURCE: Women's Univ., Nara, Japan
 SOURCE: Nippon Kagaku Zasshi (1964), 85(8), 472-3
 CODEN: NPKZAZ; ISSN: 0369-5387
 DOCUMENT TYPE: Journal
 LANGUAGE: Japanese

L20 ANSWER 163 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The effect of the addns. of Ti, Cr, V, Mn, Cu, Zn, and Ga to the catalyst was determined by measuring the elec. conductivity of finely dispersed particles. The ability of the additives to hinder the migration of H to the surface is from Cr to Cu (Cr > Ti > Mn > V > Cu). The addition of V and Mn retards the evolution of H in the same degree as Cr and Ti. The addition of accelerators creates a dumping effect on the migration of H to the surface; i.e., it prevents dehydrogenation. This fact is essential for the stable activity of the catalyst.

ACCESSION NUMBER: 1965:63041 CAPLUS
 DOCUMENT NUMBER: 62:63041
 ORIGINAL REFERENCE NO.: 62:11189f-g
 TITLE: Electric conductivity of catalytic nickel stimulated with metallic additions
 AUTHOR(S): Sokol'skii, D. V.; Matveichuk, A. Ya.
 SOURCE: Vestnik Akademii Nauk Kazakhskoi SSR (1965), 21(1), 35-8
 CODEN: VANKAM; ISSN: 0002-3213
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 164 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB cf. CA 54, 18420d. The Ni-catalyzed hydration of nitriles to amides was described. The hydration was effected in H₂O in the absence of acids and bases and was generally stopped at the amide stage. Several different types of Ni catalysts were used. One such was prepared by adding to 40 g. Zn dust in a little boiling H₂O 40 ml. of a solution containing 16 g. NiCl₂·6H₂O at 60° with vigorous shaking. A violent reaction occurred, depositing Ni on the Zn surface. Then 640 ml. 13% AcOH at 70° was added gradually with vigorous shaking, shaking was continued for 20 min., the mixture was diluted with cold water, the acid solution was decanted, and the residue was washed several times with H₂O until the washings were neutral to litmus. The catalyst thus obtained contained about 3 g. Ni. It was used directly for reactions in H₂O; it was collected by filtration and pressed before use in other solvents. The hydration of PhCN to PhCONH₂ illustrates the general procedure. Ni catalyst (4 g.) and 5 g. PhCN was refluxed 8 hrs. in 80 ml. H₂O, the catalyst filtered from the hot solution and washed with hot EtOH and H₂O, and the filtrate evaporated to yield 78% PhCONH₂, m. 127-8° (H₂O). Other solvents, MeOH, EtOH, BuOH, and dioxane, gave lower yields and side reactions; other catalysts, such as Cu, reduced Ni, and NiO-CuO, gave lower yields. With substituted benzonitriles, electron-releasing substituents retard the reaction. No hydration occurred when p-OH, o- and p-CO₂Et, and p-CHO groups were present. Metal complexes were formed when p-OH, p-NO₂, o- and p-CO₂Et, and p-CHO groups were present. Amides were also formed from aliphatic nitriles, but the yields were lower.

ACCESSION NUMBER: 1965:15189 CAPLUS
 DOCUMENT NUMBER: 62:15189
 ORIGINAL REFERENCE NO.: 62:2735b-d
 TITLE: Organic catalytic reactions. II. Hydration of nitriles to amides with Ni catalysts
 AUTHOR(S): Vatanabe, Kenichi
 CORPORATE SOURCE: Metropol. Univ., Tokyo
 SOURCE: Bulletin of the Chemical Society of Japan (1964), 37(9), 1325-9
 CODEN: BCSJAB; ISSN: 0009-2673
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 62:15189

L20 ANSWER 166 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB A hydrazine nitrate (I) based powder having good phys. strength, high explosive power, and impact sensitivity >10 lb.-in., which will burn only when in contact with flame, is provided by curing a mixture containing 50-80% powdered I, 13-40% binder such as a polyester derived from polyhydric alcoh. and polybasic acids and having a mol. weight of 500-5000, and 3-10% curing agent such as the diglycidyl ether of bisphenol A or F at 100-150°F. Powdered metals (5-15%), such as Mg, Al, Ti, Si, Zr, Sn, Zn, and Cu, and minor proportions of burning rate modifiers, such as (NH₄)₂Cr₂O₇, modifiers such as polyethylene glycol and polypropylene glycol, curing rate modifiers such as Mg stearate, and curing catalysts, such as 4,4'-methylenebis(2-chloroaniline), n-phenylenediamine, and tris(dimethylaminoethyl)phenol, may be present also. Thus, a polyester 16.05 (prepared from 9.4 moles adipic acid and 8.94 moles (HOCH₂CH₂)₂O, acid number 60, viscosity 90 poises at 80°F.), Al powder 12, diglycidyl ether of bisphenol A 3.95, 4,4'-methylenebis(2-chloroaniline) 1.5, Mg stearate 0.5, and (NH₄)₂Cr₂O₇ 1 part were mixed for 15 min., 65 parts I added, mixing continued 1 hr., and the mixture cured for 96 hrs. at 120°F. The product had a tensile strength of 5 lb./in.², strain 0.1 in./in., modulus 71 lb./in.², penetration of mild steel when cast in 4 in. lengths of 1 in. pipe 0.186 in., and impact sensitivity 13.8 lb.-in.

ACCESSION NUMBER: 1965:2470 CAPLUS
 DOCUMENT NUMBER: 62:2470
 ORIGINAL REFERENCE NO.: 62:395d-e, 396a-b
 TITLE: Castable explosive composition
 INVENTOR(S): Vriesen, Calvin W.
 PATENT ASSIGNEE(S): Thiokol Chemical Corp.
 SOURCE: 3 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3155552		19641103	US	19610308

L20 ANSWER 165 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The reaction of H, a primary or secondary alc., and the diethylene ethers YCH₂CH₂OCH₂CH₂Y' (I) gave the title compds. (II). The catalyst contained Ni, Co, Cu, and oxides of Cr, Ti, Th, Mg, Zn, Mn, and the rare earths. In a 1400-ml. rocking autoclave were placed 158 g. diglycolamine (II) (Y = Y' = NH₂, R = H) (III), 130 g. EtOH, and 75 g. Ni-Cu-Cr catalyst. The autoclave was flushed twice with H, added to 500 psig., and the mixture heated 3 hrs. at 220° and 1500 psig., and filtered. In addition to recovered III and EtOH, 1 g. morpholine and 101 g. N-ethylmorpholine were obtained. Similarly prepared were the following II (alkyl given): Me, isooctyl, b10 115-30°.

ACCESSION NUMBER: 1965:9155 CAPLUS
 DOCUMENT NUMBER: 62:9155
 ORIGINAL REFERENCE NO.: 62:1670a-c
 TITLE: N-Alkylmorpholines production
 INVENTOR(S): Advani, Prem S.; Speranza, George P.
 PATENT ASSIGNEE(S): Jefferson Chemical Co., Inc.
 SOURCE: 5 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3151113		19640929	US	19620219

L20 ANSWER 167 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Irradiation generally increases and in few cases decreases the activity of surfaces. The catalytic activity of Cu or ZnO is reduced by γ-irradiation, while that of silicagel or Ni single crystals is increased. On the other hand, the oxidation of Cu is enhanced by n irradiation, probably because of lattice defects and not of O activation. The increased oxidation of graphite in air or O during n or γ-irradiation is due to lattice defects as well as to O₃ formed upon irradiation. In electrolyte solns., the puzsivation of Al is accelerated by irradiation. As regards potential changes, no uniformity of results could be obtained. Irradiation of plastics can produce undesirable (cross linking, destruction) and desirable results: by block polymerization of Teflon with monomers, a dyeable product was obtained, while irradiated polyethylene could be given a printing.

ACCESSION NUMBER: 1964:481400 CAPLUS
 DOCUMENT NUMBER: 61:81400
 ORIGINAL REFERENCE NO.: 61:14152h, 14153a
 TITLE: Effect of nuclear radiation on the surface of construction materials
 AUTHOR(S): Neider, R.
 SOURCE: Umschau Wiss. Tech. (1964), 64(17), 530-3
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1351709		19640207	FR	
PRIORITY APPL. INFO.:			GB	19620221

ACCESSION NUMBER: 1964:444581 CAPLUS
DOCUMENT NUMBER: 61:44581
ORIGINAL REFERENCE NO.: 61:77477-h
TITLE: Catalytic action of cations on the hydrolysis of
disulfates
AUTHOR(S): Thilo, Erich; Lampe, Fred v.
CORPORATE SOURCE: Deut. Akad. Wiss., Berlin
SOURCE: Ber. (1964), 97(7), 1775-82
DOCUMENT TYPE: Journal
LANGUAGE: Ungaviable

continued for 5 h. After standing at room temperature for 14 h., the mixture was cooled to 0°, 19.4 g. AcCHCH₂(OMe)₂ added over 1 h., the resulting mixture stirred at room temperature for 4 h., treated with 100 mL. 2N H₂SO₄ at 0°, and extracted with Et₂O. A 10-g. portion of the acetylenic diol acetal obtained from the Et₂O extract was hydrogenated in the presence of 5% Pd-C and 0.5 g. quinoline in MeCOEt. Refluxing 6.5 g. of the reduced compound with 0.7 g. C₅H₅N and 0.75 mL. concentrated HCl in MeCOEt gave VII which was further purified by chromatog. on Na Al silicate. X was prepared by treating a solution of 5 g. all-trans-vitamin A aldehyde in 30 mL. CHCl₃ and 2 mL. MeOH with 3.15 g. N-bromosuccinimide in 150 mL. CHCl₃ and 4 mL. MeOH for 5 min. at 0°, adding 7.5 g. N-ethylmorpholine, and allowing the mixture to warm to room temperature over a 3-h. period. The mixture was then diluted with 300 mL. Et₂O, washed with 10% HCl, 0.5N KOH, and H₂O, dried (Na₂SO₄), evaporated, dissolved in 100 mL. petr. ether, and chromatographed on 350 g. Doucil to give 3.04 g. X (yellow zone eluted with Et₂O). X₁ was prepared similarly by replacing MeOH with Me(CH₂)₁₁OH. XII was prepared from a mixture (XIII) of 4-methoxy- α -ionone and 4-methoxy- β -ionone in the

ACCESSION NUMBER: 1964:432649 CAPLUS
DOCUMENT NUMBER: 6132649
ORIGINAL REFERENCE NO.: 61:5702e-h, 5703a-d, 5704a
TITLE: Polyene compounds from thiapyrans
CHEMAB: Albert C. J. Roseson, Charles D.
PATENT ASSIGNEE(S): Eastman Kodak Co.
SOURCE: 14 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATEX INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3125571		19640317	US	19620528
FR 1378802			FR	

L20 ANSWER 171 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Primary amines were prepared from an aliphatic alc. and NH₃ in the presence of a special catalyst consisting of a combination of 3 metallic components, Ni, Cu, and an oxide of Cr, Ti, Th, Zn, Mg, in a ratio of 50-90%; 90-50% and .apprx.0.5-5%, resp. Thus, in a stirred autoclave 666 g. BuOH and 200 g. catalyst containing 78.1 weight-% Ni, 14.2 weight-% Cu, and 1.06 weight-% Cr as Cr₂O₃ was flushed twice with H₂, 459 g. liquid NH₃ added, H added, and the mixture heated to 207° in 45 min., heated 2 hrs. at 207-13°/2200-2350 lbs./in.², and worked up to give 100 g. unreacted BuOH, 340 g. BuNH₂, and 140 g. Bu₂NH. Other examples were given.

ACCESSION NUMBER: 1964:403950 CAPLUS
DOCUMENT NUMBER: 61:3950
ORIGINAL REFERENCE NO.: 61:574d-e
TITLE: Primary amines
INVENTOR(S): Shirley, Robert I.; Speranza, George P.
PATENT ASSIGNEE(S): Jefferson Chemical Co., Inc.
SOURCE: 3 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3128311	---	19640407	US	19611211

L20 ANSWER 173 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Alkylarenes are treated with NH₃ in the presence of a refractory material containing ions of a heavy metal (which is capable of forming a Werner amine complex), such as Cr, Co, Ni, Cu, Zn, Pd, Ag, or Pt to give nitriles. Thus, 13 X-type, Na aluminosilicate mol. sieve is treated with ZnCl₂ solution to give a zeolite containing 57% Na and 43%

Zn and a PhMe-NH₃ mixture, NH₃-PhMe molar ratio 13:1, is contacted with the prepared catalyst 2-4 sec. at 583° to give benzotrile, 73% yield per pass. Similarly prepared is p-tolunitrile with a small amount terephthalo nitrile.

ACCESSION NUMBER: 1964:90610 CAPLUS
DOCUMENT NUMBER: 60:90610
ORIGINAL REFERENCE NO.: 60:15790g-h
TITLE: Aromatic nitriles
INVENTOR(S): Jones, Daniel G.; Landis, Phillip S.
PATENT ASSIGNEE(S): Socony Mobil Oil Co., Inc.
SOURCE: 21 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1350206	---	19640124	FR	19620202
GB 956892	---	---	GB	---
US 3231600	---	1966	US	---

L20 ANSWER 172 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB Since in the conversion of CH₄ to H₂ + CO over Ni catalysts, the maximum S content that can be tolerated is 3 mg./cu. m. for a tubular furnace and 12-20 mg./cu. m. for a "mine-shaft" reactor, expts. were carried out on the desulfurization of shale gas from the Slaty Combine with a normal level of 160-200 mg./cu. m. As an absorbent for organic S. compds., 2 grades of ZnO were tried, one prepared

by the decomposition of ZnCO₃ at 350° (I) and the other by high-temperature roasting of Zn ores (II). Based on total organic S in the shale gas, the thiophene S ranged from 6 to 35%; CS₂, 10-29%; and COS 40-65%. Since thiophene is not absorbed by ZnO, the gas was first passed over activated birch charcoal. At 380-400° and a liquid hourly space velocity (LHSV) of 120-210, the S was reduced to 4-14 mg./cu. m., the lower amts. being obtained by the use of I. Samples of spent I on regeneration decline to the activity of II. CO₂ and unsatd. hydrocarbons should be removed before reforming shale gas. On a laboratory scale, using

a quartz reactor and Ni catalyst (GIAP-3), desulfurized shale gas, without CO₂ and olefin removal, was reformed at 800° at a volume ratio of steam to gas of 1.1: 1.0 and a LHSV of 350. The converted gas on a dry basis had the following average composition: CO₂, 10.4; O, 0.8; CO, 18.4; H₂, 57.4; CH₄, 3.2; and N₂, 9.8% by volume. The consumption of steam by reaction with hydrocarbons was 0.4 volume per volume of original gas. The com. catalyst used converted CH₄ with an excess of steam of 100-120%. Incomplete conversion was attributed to only partial reduction of the catalyst, some of the Ni being left in the spinel form. The increase in volume of gas at complete CH₄ conversion was 220%.

ACCESSION NUMBER: 1964:403484 CAPLUS
DOCUMENT NUMBER: 61:3484
ORIGINAL REFERENCE NO.: 61:499d-g
TITLE: Catalytic conversion of shale gas with steam
AUTHOR(S): Sharonov, G. E.; Pegushin, A. V.; Pyl'nikova, L. V.
SOURCE: Tr. Vses. Nauchn.-Issled. Inst. po Pererabotke i Ispol'z. Topliva (1963), No. 12, 174-80
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L20 ANSWER 174 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB The effect of 160 inorg. and organic compds. present in 0.1-0.15% concns. on autoxidn. of furfural (I) at room temperature was followed colorimetrically. Pos. catalytic action of acids depended on their degree of dissociation and solubility in I, and decreased in the order HCl > H₂SO₄ > HNO₃ > CCl₃CO₂H > HCO₂H > AcOH > H₃BO₃. Sparingly soluble succinic, maleic, citric, and α-oxoglutaric acids increased the autoxidn. rate very little, but fumaric and phthalic acids acted as inhibitors. Catalytic initiation was observed in the presence of acidic salts KHSO₄, NH₄OH.HCl, (NH₄)₂SO₄, PhNH₂.HCl, PhNH₂.H₂SO₄, but KHSO₄ and NaHSO₄ showed an inhibiting action. Certain Fe(II), Fe(III), Cu, Mn, Zn, Al, and NH₄ salts exhibited pos. catalytic action as well. The catalytic action of acidic compds. might be explained by the addition of H⁺ to O atom in the furan ring to yield a pos. ion with a very reactive conjugated double bond system. The almost identical catalytic effect of Li, K, Na, and Ca hydroxides, slightly pos. in the beginning, then strongly inhibiting, was attributed to the neutralization of pos. acidic catalysts formed during I autoxidn. The inhibiting action of aromatic compds. decreased in the order -OH > NH > -OMe > -NHMe₂ > -Me > -H > -NH₂. Water in concentration <3% as well as small amts. of alcs. and ethers usually present in tech. I did not affect its autoxidn. rate.

ACCESSION NUMBER: 1964:82239 CAPLUS
DOCUMENT NUMBER: 60:82239
ORIGINAL REFERENCE NO.: 60:14343f-h
TITLE: Investigation of autoxidation of compounds related to furan. VI. Catalytic effect of organic and inorganic compounds on autoxidation of furfural
AUTHOR(S): Chernyaeva, G. N.; Khol'kin, Yu. I.
SOURCE: Issled. v Obl. Khim. i Khim. Tekhnol. Drevesiny, Akad. Nauk SSSR, Sibirsk. Otd., Inst. Lesa i Drevesiny (1963) 38-54
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L20 ANSWER 175 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB For the production of higher alcs., a suitable crude material is represented by the product obtained by the condensation of CO and H over fused iron catalysts; impurities include carbonyl compds. (up to 20%), esters (up to 5%), organic acids (not >3%), and hydrocarbons (up to 1%). Non-alc., O-containing impurities and olefins can be removed by selective catalytic hydrogenation. For this purpose 3 different catalysts were tested: Ni-Cr (I), Ni-Cr (II), and Cu-Cr (III). Hydrogenation expts. were conducted in a flow-type reactor, 16 mm. internal diameter, of alloy steel, containing 50 cc. of 3-7 mm. catalyst granules over which was placed a layer of powdered glass. I was prepared from a mixture of 65-58% ZnO, 17-19% Cr2O3, 7-9% CrO3, and 5-8% H2O, by reduction at 100 atmospheric at 400° with a mixture of 75% H-25% N, space velocity 3000 hr.-1, for 4 hrs.; II by reduction of a mixture of 26% Ni2O3, 65% Cr2O3, and 9% H2O, with the same H-N mixture at 50 atmospheric and 280°, space velocity 3000 hr.-1, for 3 hrs.; and III by reduction of a mixture of 50% CuO, 43% Cr2O3, 2% graphite, and not >8% H2O with the H-N mixture at 300°, space velocity 3000 hr.-1, for 3 hrs. With I the original crude stock had an acid number of 27, ester number 9, hydroxy, number 323, carbonyl number 89, and iodine number 11 (Feeds to the other 2 catalysts were similar but not identical). Increase in temperature of hydrogenation over the range 260-360°, while maintaining pressure at 320 atmospheric, increased the rate of hydrogenation but at temps. >320° some hydrogenation of alcs. to hydrocarbons took place. The reaction is also accelerated by increase of pressure from 100 to 200 atmospheric, while lesser, proportional, increases of rate result from increasing the pressure to 320 atmospheric, which represents an optimal pressure for selective hydrogenation to alcs. The space velocity of liquid charge was varied from 0.5 to 3.0 hr.-1 and that of the H-N mixture from 600 to 4800 hr.-1. An increase of space velocity of the liquid feed at constant rate of N-H feed leads to some decrease in the degree of conversion of carbonyl compds. to alcs. Increase in the space velocity of the H-N mixture at constant liquid charge rate is accompanied by a decrease in the proportion of secondary products (e.g., olefins from alcs.) and an increase in alc. yield. Only at very large excesses of H are secondary reactions (especially paraffinic hydrocarbon production) accelerated. Optimal space velocities are 1 and 1200 hr.-1, resp., for liquid feed and N-H mixture, although these rates can be simultaneously doubled with little or no loss in hydrogenation efficiency. I was used for 160 hrs. continuously without loss of activity and without powdering. I is effective in completely converting organic acids to alcs., but esters are converted only at temps. of 340-60°. The degree of conversion of carbonyl compds. never exceeds 90%, except at temps. >360°, when hydrogenation of alcs. to saturated hydrocarbons and dehydrogenation to olefins suddenly become serious secondary reactions. In expts. with II, higher activity than that of I was manifested, and the optimal temperature was 150°. Carbonyl compds. over this catalyst are 100% converted to alcs. at temps. >140°. Unsatd. compds. are completely absent from the hydrogenates except at temps. >940°, when they are produced by dehydrogenation of alcs. Esters are not effectively removed by II and actually at 240° the ester content of the product is greater

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 AB A resin is prepared by heating at >300°F. a mixture of 1-6 parts by weight of a sorbitan mono fatty acid ester with 1 part by weight of an ester of stearic acid and an alc. having 1-6 C atoms in the presence of substantially pure Zn in a blown oil solvent to effect polymerization. A polar organic corrosion inhibitor is dispersed during polymerization. A mixture of 75% Zn and 25% Pb, a 50-50 Zn-Cu liner, and a pure Cu liner gave no polymerization. The polymer composition had rubberlike characteristics and high gloss. Upon application to steel, it formed an anticorrosive film resistant to hot salt water. U.S. 3,093,492 Appl. May 27, 1960; 3 pp. Besides the reactants of the preceding patent, 1 part by weight BzOH or its derivs. is used in the resin-forming mixture
 ACCESSION NUMBER: 1963:436464 CAPLUS
 DOCUMENT NUMBER: 59:36464
 ORIGINAL REFERENCE NO.: 59:6619d-f
 TITLE: Resinous metal coating compositions
 INVENTOR(S): Larsen, Norman B.
 PATENT ASSIGNEE(S): Corrosion Reaction Consultants, Inc.
 SOURCE: 2 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3093491		19630611	US	19600505

L20 ANSWER 175 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN (Continued)
 than that of the feed, because of the catalysis of esterification reactions. Org. acids require temps. >200° for complete conversion to alcs. over II. The effects of pressure and of space velocity of feed and N-H mixt. are similar to those observed with I. After extended runs II is found to be seriously powdered. III was found to be active and selective over a broad range of conditions. Beginning at 220°, org. acids, carbonyl compds., and unsatd. compds. are hydrogenated completely. Esters are not hydrogenated, however, to more than 60% at the highest practicable temp., bearing in mind that at temps. >300° the catalyst loses selectivity and hydrogenates alcs. to hydrocarbons. Optimal pressure is 320 atm. After 150 hrs., III retained activity and powdered only slightly. Secondary or side reactions of various sorts can be suppressed to a large extent by increasing the space velocity of the H-N mixt. In general, I, II, and III respond similarly to variation in space velocities of liquid feed and H-N mixt. Of the 3 catalysts, III is preferred because of more uniform and homogeneous product compn., facilitating further fractionation and purification.
 ACCESSION NUMBER: 1964:38366 CAPLUS
 DOCUMENT NUMBER: 60:38366
 ORIGINAL REFERENCE NO.: 60:6735e-h, 6736a-d
 TITLE: Selective catalytic hydrogenation of aliphatic oxygen-containing compounds under high pressure
 AUTHOR(S): Loktev, A. M.; Kagan, Yu. B.
 SOURCE: Neftskhimiya (1963), 3(6), 892-9
 CODEN: NEFTAH; ISSN: 0028-2421
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 177 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB A supported metal catalyst consisting of either Ni, Co, Cu, Mn, Pt, Pb, Hg, Cd, Au, or Zn is produced by first converting the metal powder to the metal ammine carbonate, of the general formula 2M(NH3)xCO3, where x has a value of 1 through 6, so as to get an aqueous solution of the ammine containing 0.1-5% of the metal by weight. An inert carrier, such as kieselguhr, diatomaceous earth, or pumice is then suspended in the aqueous ammine solution such that the metal-to-carrier ratio is 1:1 to 1:2. The basic carbonate of the metal is precipitated on the suspended carrier particles by heating the suspension at 200-212°F. with five steam to evaporate NH3. After filtering, the coated particles are dried at 250°F., and then calcined at 650-850°F. to convert the basic carbonate to the metal oxide, which is partially reduced in H at 700 800°F. for 1-12 hrs. so that about 30% to 80% of the metal oxide is not reduced. By this method catalysts having initial activity of 16-20 min. and having 20 to 25 reuses can be prepared. The activity is determined by measuring the time required to reduce the n of cottonseed oil a specified amount by hydrogenation.
 ACCESSION NUMBER: 1963:432502 CAPLUS
 DOCUMENT NUMBER: 59:32502
 ORIGINAL REFERENCE NO.: 59:5840e-g
 TITLE: Catalysts
 PATENT ASSIGNEE(S): Chematron Corp.
 SOURCE: 5 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 926235		19630515	GB	
PRIORITY APPLN. INFO.:			US	19591001

L20 ANSWER 178 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The catalytic and magnetic properties of dilute atomic, atom-ion, and ionic binary systems of metallic adsorption catalysts were studied. It was established that the character of the catalytic activity of mixed metallic catalysts is determined by the electronic structure of the atoms and the ions of the active components and by the position of the particular element in the periodic system. The magnetic susceptibility of the systems investigated varied abruptly with the catalytic activity. The interdependence observed between the catalytic activity, the activation energy, and the magnetic susceptibility proves the existence of an interaction between the atoms and ions of the components of the system as well as the variation of valence-energy properties of the atomic phase. The extreme values of the activity and magnetic susceptibility of the binary systems of the atomic adsorption catalysts agree with the simplest stoichiometric ratios of the atoms and is related with spin-valence atoms and with the original surface atomic structure. For the atom-ion and ionic catalysts ratios other than stoichiometric are observed between atoms and ions and the ions of active components. These effects are conditioned by the participation of d-electrons of the metal ions together with s-electrons of the atoms as the components participate in the reaction.

ACCESSION NUMBER: 1963:406207 CAPLUS
 DOCUMENT NUMBER: 59:6207
 ORIGINAL REFERENCE NO.: 59:1133a-d
 TITLE: Electronic structure of the atoms and catalysis in mixed atomic layers
 AUTHOR(S): Zubovich, I. A.
 SOURCE: Kataliz v Vysheh Shkole, Min. Vysshago i Srednego Spets. Obrazov. SSSR, Tr. 1-go [Pervogo] Mezhvuz. Soveshch. po Katalizu (1962), 1958 (No. 1; Pt. 1), 305-12
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 180 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Tar samples from low-temperature carbonization of brown coal in Lurgi furnaces were heated for 1 hr. at 250° with 3% catalysts containing 10% Ni, Fe, Co, Cu, V, Pb, Mo, Cr, Zn, Sn, or W, resp., on a SiO₂-Al₂O₃ carrier (I). The tar was then distilled up to 191° at 20 mm. and As was estimated in the distillate and residue. Ni, Co, and V gave the best As removal from the distillates. The same expts. were carried out with Ni catalysts prepared by reduction of NiO with H₂ by thermal fusion of Ni, from a Ni-Al alloy, from Ni-Cr steel, by reduction of Ni dimethylglyoxime, from Ni on a clay, and from Raney Ni. During heating, 5 vols. of H₂ per volume of tar was passed through the tar. Only catalysts prepared by chemical reduction of Ni compds. and Raney Ni gave good results. Pure Ni, Ni₃Sn₂, and NiAs removed, resp., 88.6, 95.9, and 62.9% of the original As from the distillates. The influence of the amount of Ni, temperature, reaction time, and the presence of H₂ was determined.

About 0.02-0.04% Ni is needed to remove 80% As at 250° in 1 hr.; a rapid decrease in activity occurred when smaller amts. of catalyst were used. Decreasing the reaction time to <60 min. had the same effect. In a pilot-plant distillation column with 3 layers of NiO catalyst containing 18% Ni on I, 7220 kg. of tar was worked up by using 830 g. Ni. The distillate contained 2-7% of the original As; the catalyst after use contained 2-4% As and had the same activity as a fresh one.

ACCESSION NUMBER: 1963:19963 CAPLUS
 DOCUMENT NUMBER: 58:19963
 ORIGINAL REFERENCE NO.: 58:3240b, 3241a-b
 TITLE: Decomposition of volatile arsenic compounds on nickel catalysts during tar distillation
 AUTHOR(S): Svajgl, Oldrich
 CORPORATE SOURCE: Vyzk. Ustav pro Chem. Využití Uhlí, Chem. Zavody CSSP, Zaluž, Czech.
 SOURCE: Chemický Průmysl (1962), 12, 473-8
 CODEN: CHPUA4; ISSN: 0009-2789
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 179 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Activation energies and entropies were found for the elimination of Cl- from chloro(ethylenediaminetriacetatoacetate)cobaltate(III) catalyzed by various metal cations in aqueous solution. A mechanism is proposed which involves the interaction of both the leaving Cl- and the free carboxylate group with the catalyst. The elimination of Cl- from the conjugate acid of this cobalt(III) complex is much less susceptible to cationic catalysis.

ACCESSION NUMBER: 1963:400464 CAPLUS
 DOCUMENT NUMBER: 59:464
 ORIGINAL REFERENCE NO.: 59:62d-e
 TITLE: Catalysis of the elimination of chloride ion from chloro(ethylenediaminetriacetatoacetate)cobaltate(III) and its conjugate acid by metal cations
 AUTHOR(S): Dyke, R.; Higginson, W. C. E.
 CORPORATE SOURCE: Univ. Manchester, UK
 SOURCE: Journal of the Chemical Society, Abstracts (1963) 2788-97
 CODEN: JCSAAZ; ISSN: 0590-9791
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 181 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB cf. CA 57, 4836c. By a simple titrimetric method, the elimination of HCl from poly(vinyl chloride) suspension polymers in the presence of FeCl₃, other metal salts, metal powders, and organic bases was investigated. Al powder has no effect. Fe accelerates HCl formation in the presence of O and air, while Zn and Cu powder are strong catalysts. Mg acts as an inhibitor. AlCl₃ has no effect, while Zn, Cu, and Co salts accelerate the decomposition of poly(vinyl chloride). Amines are strong accelerators. Pb stearate and phenyl glycidyl ether increase the induction period; Bu₂SnO has a similar effect. Bu₂SnS is a much better inhibitor. The butyltin dodecylmercaptides are also excellent inhibitors. A theory is developed in which HCl reacts with the S-bearing inhibitor to give a free mercaptan, which in turn inactivates free radicals and heavy-metal catalysts.

ACCESSION NUMBER: 1962:470004 CAPLUS
 DOCUMENT NUMBER: 57:70004
 ORIGINAL REFERENCE NO.: 57:13982b-e
 TITLE: Stability of poly(vinyl chloride) suspension polymers. II. Influence of impurities and stabilizers on the evolution of hydrogen chloride by poly(vinyl chloride) on heating
 AUTHOR(S): Rieche, A.; Grimm, A.; Muecke, H.
 SOURCE: Kunststoffe (1962), 52, 398-400
 CODEN: KUNSAV; ISSN: 0023-5563
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 182 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB A series of measurements of the catalytic recombination coefficient γ for recombination of O atoms on various metallic surfaces was made. A mol. beam and a new type detector for calibration of the atomic O content of the beam were used. Previous results are in general qual. agreement with present theories of the electronic basis of catalysis; the variation in the data is somewhat less than that reported elsewhere. 88 references.

ACCESSION NUMBER: 1962:466241 CAPLUS

DOCUMENT NUMBER: 57:66241

ORIGINAL REFERENCE NO.: 57:131911,13192a

TITLE: A study of oxygen recombination on metallic surfaces by means of an atomic beam

AUTHOR(S): Hoening, Stuart Alfred

CORPORATE SOURCE: Univ. of California, Berkeley

SOURCE: United States Department of Commerce, Office of Technical Services, PB Report (1960), 157,489, 154 pp.

CODEN: XCPRAL; ISSN: 0099-8567

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

L20 ANSWER 183 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB The influence has been tested of 17 substances on the rate of reesterification of 15 g. di-Me dehydromucate by 15 cc. HO(CH₂)₂OH at 195° and on the following condensation at 245°, which was completed at 1-2 mm.; the time of condensation was 4 and 2 hrs., resp. From the substances tested (punice, Na₂CO₃, Zn(AcO)₂, Zn borate, (iso-PrO)₃Al, B₂O₃, PbO, Sb₂O₃, Mn(II)borate, Co(II) borate, Mg, Zn, Al, Ni, Cu, Fe, and rustless steel), the best results were obtained with 0.03-0.054 weight % borate, where light-colored polymers (I) with inherent viscosity of approx. 0.6 were produced. In the presence of Fe or Cu, colored I were obtained, hence Cu and Fe cannot be used as construction materials. At 250° decomposition, of I occurred regardless of the catalyst used.

ACCESSION NUMBER: 1962:463489 CAPLUS

DOCUMENT NUMBER: 57:63489

ORIGINAL REFERENCE NO.: 57:12701h-i

TITLE: Catalysts for the preparation of the polyethylene derivative of dehydromucic acid

AUTHOR(S): Zamorsky, Zdenek

SOURCE: Chemicky Prumysl (1961), 11, 387-90

CODEN: CHPUA4; ISSN: 0009-2789

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

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AB cf. CA 55, 20941b. At pH 8.6, the optimum pH for the catalytic effect, glycyDL-leucine is measurably hydrolyzed at 37° and after 6 hrs. in the presence of Th⁴⁺, Zr⁴⁺, and Er³⁺; after 24 hrs. in the presence of Ho³⁺ and Yb³⁺; and after 72 hrs. in the presence of Dy³⁺, Nd³⁺, Eu³⁺, Gd³⁺, Sm³⁺, and Hf⁴⁺; at 70° after 24 hrs. by Tb³⁺, Bi³⁺, Fe³⁺, Fe²⁺, Zn²⁺, Co²⁺, Cd²⁺, y³⁺ Mn²⁺, and Cu²⁺; and after 48 hrs. by Pb²⁺, Ni²⁺, Cr³⁺, W⁶⁺, In³⁺, Ti³⁺, and V⁴⁺. At 70° and after 72 hrs. the alkali metal ions show LO activity. Neither do Be²⁺, Mg²⁺, Ca²⁺, Sr²⁺, Ba²⁺, Al³⁺, Ga³⁺, Tl³⁺, Ge⁴⁺, Sn²⁺, Ag⁺, MoO₄²⁻, Pd²⁺, and PtCl₄²⁻. The effectiveness of the catalyst is a function of an unstable reaction product between catalyst and the electron pair of the amino or imino N or of the carbonyl O. Decomposition of polypeptides is discussed in terms of the nucleophilic reagent, electrostatic effects, inductive effects of the carboxylate ion, and the steric effect of the side chain.

ACCESSION NUMBER: 1962:433408 CAPLUS

DOCUMENT NUMBER: 57:53408

ORIGINAL REFERENCE NO.: 57:66622-i

TITLE: Cation-catalyzed decomposition of the CONH group in peptides and N-acetyl amino acids

Bamann, Eugen; Haas, Johann George; Trapmann, Heinz

CORPORATE SOURCE: Univ. Munich, Germany

SOURCE: Arch. Pharm. (1961), 294, 569-80

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

L20 ANSWER 185 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN

AB cf. ibid. 11, 153(1958); CA 56, 12348c. The rate of oxidizing Et₂NH with K₂S₂O₈ was studied in the presence of Zn, Cd, Co, Cu, and Ag. In the presence of Zn²⁺ and Cd²⁺ the rate of oxidation was reduced whereas in the presence Co²⁺ and, particularly of Cu²⁺ and Ag⁺, it was accelerated. The reaction was 2nd order. The rate of reaction was directly proportional to the 1st order of concentration of catalyst ions and amines. The catalytic action of Cu²⁺ in the peroxydisulfate decomposition in the alkaline solution was higher than that of Ag⁺, whereas in the presence of amines it was lower. This can be attributed to the presence of higher amount of ligand mols. in the Co-amine complex than in the Agamine complex so that formation of kinetically active complexes in the first case is more difficult than in 2nd. The low catalytic activity of Co²⁺ is attributed to the low rate of oxidation (Co²⁺ + S₂O₈²⁻) and to the higher stability of the oxidation product (Co³⁺ as compared to Cu²⁺ and Ag⁺ and higher stability of Co³⁺ amines in comparison with those of Co²⁺. 31 references.

ACCESSION NUMBER: 1962:400631 CAPLUS

DOCUMENT NUMBER: 57:631

ORIGINAL REFERENCE NO.: 57:1031,104a-c

TITLE: Kinetics of the reaction of potassium peroxydisulfate with amines in aqueous solutions. IX. Effect of cations on the kinetics of the peroxydisulfate-diethylamine reaction

Chaltykyan, O. A.; Beileryan, N. M.

Izvest. Akad. Nauk Armyan. S.S.R., Khim. Nauki (1961),

14, 197-207

DOCUMENT TYPE: Journal

LANGUAGE: Russian

L20 ANSWER 186 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The title dehydrogenation was carried out in the presence of a Cu catalyst activated with chromates. Cu tetramine salts were mixed with Na or NH₄ chromate and Na₂CO₃ or Zn salts, decomposed thermally, calcined at 600-800° and reduced at 200-300°. The weakly alkaline catalyst contained 0.5-3.0 parts Cr₂O₃/97-99.5 parts CuO. Thus, a solution of 480 g. Cu nitrate in 300 ml. H₂O was treated successively with 750 g. NH₄OH (d20 0.91-0.93) and a solution of 5.02 g. Na₂CrO₄.4H₂O and 1.62 g. Na₂CO₃ in 25 ml. H₂O. Sieved pumice (grain size 2-4 mm.) (600 ml.) was heated in vacuo and treated with the above solution which was preheated at 70-80°. The impregnated pumice was dried at 100°, decomposed thermally at 400°, calcined at 600-800° 2 hrs., and reduced in a 1:1 H-N atmospheric. The impregnation was repeated twice with the above solution. The ready catalyst contained 40.09% CuO, and 0.53% Cr₂O₃. The vapors of 125-35 ml. 94% EtOH were passed at 300-80° through a tube filled with 0.5 l. (282 g.) catalyst. After one passage 62-72% EtOH had been dehydrogenated at 95.3-99.3% to a mixture of MeCHO 90-3, EtOAc 3-4, HOAc 1.1, heavy hydrocarbons 0.3, CO₂ 0.7, and CO 0.2 mole-%. Similarly, BuOH gave a yield of >90% PrCHO. The catalyst could be regenerated.

ACCESSION NUMBER: 1962:66579 CAPLUS
 DOCUMENT NUMBER: 56:66579
 ORIGINAL REFERENCE NO.: 56:12744a-d
 TITLE: Aldehydes by dehydrogenation of low-molecular-weight primary aliphatic alcohols
 INVENTOR(S): Opitz, Wolfgang; Urbanski, Werner
 PATENT ASSIGNEE(S): Knapsack-Griesheim A.-G.
 SOURCE: Addn. to Ger. 1,097,969 (CA 55, 25757g)
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1103317		19611012	DE	19550208

L20 ANSWER 187 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN (Continued)
 yielded 104 material, m. 93-100°, sublimed at 70-5°/0.1 mm. to yield 2-naphthol-1-acetic acid lactone, m. 104-5°, λ 5.58 μ , λ 230,270, 281,294, 316,323, 331 μ [e 60,000, 3000 (2 triplets)]. No other cryst. material was obtained from the chromatogram. Anhyd. Me₃COH (10 ml., b. 82.3° distd. from CaH₂) stirred with 94 mg. freshly cut K (N atm.) at 100° treated with 599 mg. II, m. 53-4°, in 10 ml. anhyd. Me₃COH, the light yellow anionic mixt. treated dropwise 15 min. with 160 mg. freshly distd. H₂C=CHCN, b. 76.5°, in 5 ml. Me₃COH and kept 9 hrs. at 25° (N atm.), neutralized with slight excess of AcOH, soln. dild. with H₂O, extd. with CHCl₃, and the oily product triturated with Et₂O yielded 72.5% 5-(β -cyanoethyl)-2,3,4-trimethoxy-6-benzocycloheptanone (IX), m. 110.5-112°, λ 4.45, 5.90 μ . IX (395 mg.) refluxed 24 hrs. with 6 g. KOH in 36 ml. 1:2 MeOH-H₂O, the mixt. washed with Et₂O, the aq. soln. acidified with HCl, extd. with Et₂O, the acidic product (407 mg.) heated (N atm.) 9 hrs. at 100° with 20 mg. NaOAc in 20 ml. distd. Ac₂O, b. 136-7°, the solvent distd. at 80° in vacuo (N atm.), the residue taken up in Et₂O, the washed and dried soln. evapd., the product (264 mg., m. 99-103°) filtered in 1:19 Me₂CO-petr. ether through 3.0 g. Florisil, eluted with petr. ether, and the fraction (221 mg.), m. 103.3-4.6°, recrystd. from petr. ether gave 2,3,4-trimethoxybenzocyclo-5-hepten-6-ol-5-propionic acid α -lactone (X), m. 104.0-5.2°, λ 2.62 μ (e 14,000), 5.74, 6.02 μ , n.m.r. peaks τ -75, 36, 80, 101 cycles/sec. relative to H₂O. X was obtained in another cryst. form, m. 89.0-90.5°, by rapid crystn. at 0° from petr. ether. Treatment of the crude oxo acid with C₆H₁₁N: C: NC₆H₁₁ in C₅H₅N gave an N-acetyl urea, m. 123-5°, λ 3.0, 5.9, 6.05-6.10 μ . IX (16.558 g.) in 75 ml. dry C₆H₆ stirred with gentle refluxing with 75 ml. anhyd. Et₂O, 25 g. activated Zn, a few crystals of iodine, and 1 l. g. freshly distd. BrCH₂CO₂Me, b. 143°, the mixt. gradually treated with 175 g. Zn and 23 g. BrCH₂CO₂Me in 90 min., stirred 1 hr., an agglutinated mass refluxed 2 hrs., dild. with excess AcOH and a small amt. of MeOH, the soln. decanted, extd. with H₂O, the H₂O extd. with C₆H₆, the combined org. layers evapd., the residue freed from AcOH by azeotropic distn. with PhMe, the cryst. residue chromatographed on 350 g. Al₂O₃ (Merck), carefully eluted with 1:9 Me₂CO-petr. ether, and the fractions triturated with Et₂O yielded 18.8% trans-6-carbomethoxy-methyl-5-cyanoethyl-2,3,4-trimethoxybenzo-6-cycloheptanol (XI), m. 145.0-5.8° (Me₂CO-petr. ether), λ 2.85, 4.45, 5.85 μ , and 87.6% cis-6-carbomethoxymethyl-5-cyanoethyl-2,3,4-trimethoxybenzo-6-cycloheptanol (XII), m. 118.919.41 X 2.88, 4.45, 5.85 μ . Further elution with 1:3 Me₂CO-petr. ether gave 0.550 g. ester lactone (XIII), corresponding to XII. On sapon. with refluxing KOH in MeOH XII and XI were converted to the corresponding hydroxy dicarboxylic acids (XIV, XV), m. 83-8° and 206-8°, resp. The hydroxy diester from XIV (51.5 mg., m. 99-101°) and 2 mg. p-MeC₆H₄SO₃H₂O heated 8 hrs. at 90° in 2.5 ml. Ac₂O, dild. with 30 ml. CHCl₃, the soln. shaken with 104 aq. NaHCO₃, the aq. layer extd. with CHCl₃, the washed and dried CHCl₃ ext. evapd., and the product chromatographed in Me₂CO-petr. ether on Al₂O₃ gave 20 mg. material, m. 90-100°, recrystd. from petr. ether to give Me 6-acetoxy-6-carbomethoxymethyl-2,3,4-trimethoxybenzocycloheptane-5-propionate, m. 100.3-1.1° λ 5.8 μ , also prepd. by treatment of the hydroxy diester with H₂C=CHMeOAc and acid catalyst. XIV (from hydrolysis of 2.216 g. XII) in 15 ml. C₅H₅N kept 2 days at 25° with 1.262 g. C₆H₁₁N: C: NC₆H₁₁, the cryst. product washed with Et₂O, the C₅H₅N and Et₂O solns. evapd. in a stream of N, the residue dried 24 hrs. in vacuo, taken up in 100 ml. MeOH, methylated with CH₃N₂ in Et₂O, the residue on evapn. (2.45 g.) chromatographed on 100 g. Al₂O₃, eluted with

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 AB Synthesis of colchicine (I) involved transformation of 2,3,4-trimethoxybenzocycloheptan-6-one (II) into the previously unknown key intermediate, deacetamidocolchicine (III) into which the required NHAc group might be introduced subsequently. In connection with attempts to use II in simple alkylation and Michael-type reactions, model expts. involving the more available 1,2,3,4-tetrahydronaphthalen-2-one (IV) were carried out. Na (1 g.) in 300 ml. anhydrous MeOH (ice bath) stirred magnetically with 5.0 g. IV (b11 130-5°), the mixture stirred 5 min. with dropwise addition of 4.8 g. AcCH:CHCO₂Et (b7 80-90° n20D 1.4506), stirred 40 min. at 25° before dilution with 600 ml. H₂O, kept 30 min., and filtered gave 6.10 g. material, m. 190-5°, recrystd. from MeOH to yield 48.5% 5,7-benzo-4-carbomethoxy-2-hydroxy-2-methyl-9-oxobicyclo[1.3.3]nonane (V), m. 200.0-2.5° (decomposition), λ 2.95, 5.75, 5.85 μ (KBr), λ 266 μ (e 500). The mother liquors yielded a few mg. of a possible C-2 epimer, m. 163-5° IV refluxed 3 hrs. with excess purified pyrrolidine, b. 86.5-7.0° in C₆H₆ under a Dean-Stark head gave IV pyrrolidine enamine (VI), m. 81-2°. VI (1 g.) and 0.72 g. AcCH:CHCO₂Et, b7 80-90°, stirred 14 hrs. at 25° in 25 ml. dry C₆H₆, the solvent removed in vacuo, the residue refluxed with 1 g. NaOAc in 60 ml. 1:1 10-AcOH-H₂O-MeOH, the MeOH evaporated, the dark green solution diluted with 50 ml. H₂O, the tarry product triturated with absolute alc., and the crystalline product recrystd. from MeOH gave a small yield of V. V (1.00 g.) in 5 ml. absolute alc. heated 3 hrs. with occasional swirling at 60° with 3 ml. freshly distilled HC(OEt)₃, b. 144-5°, containing 1 drop concentrated H₂SO₄, the cooled solution treated with excess Na₂CO₃, the filtrate and alc. washings evaporated, the residue triturated with MeOH, and slowly crystallized gave a mixture of 2 dehydrated ketals. Modification of the procedure by heating the mixture 10 min. at 80°C yielded 10-20% 5,7-benzo-4-carbomethoxy-9,9-diethoxy-2-methylbicyclo[1.3.3]nonane (VII), m. 87.6-8.7° (MeOH), λ 5.85, 6.12, 11.18 μ , nuclear magnetic resonance (n.m.r.) peak at 65 cycles/sec. relative to C₆H₆. The same reaction run at 60° 6 hrs. yielded 10-25% 5,7-benzo-4-carbomethoxy-9,9-diethoxy-2-methylbicyclo[1.3.3]non-2-ene (VIII), m. 111.6-12.8° λ 5.85 μ . Acid hydrolysis of VII and VIII to the corresponding ketones failed. VII (90.6 mg.) in 10 ml. AcOH ozonized 40 min. at 25°, the mixture steam distilled with collection of 350 ml. distillate (ice bath) with addition of 148 mg. dimedon in 5 ml. alc. at the half-way point, the distillate adjusted to pH 4.5 with dilute aqueous NaOH, and kept 16 hrs. at 0° yielded 44.7% HCHO dimedon deriv., m. 187.59.5°. VIII [5.1 mg., λ 220 μ (e 4600, absolute MeOH)] and 0.23 g. Na in 100 ml. absolute MeOH kept 2 hrs. at 25° gave a mixture, λ 220 μ (e 10,500, NaOMe in MeOH), ascribed to appearance of the isomeric α,β -unsatd. ester. V (1.0 g.) and 2 drops concentrated H₂SO₄ refluxed in 25 ml. H₂C=CHMeOAc, b. 95-6°, the homogeneous yellow solution evaporated in vacuo, and recrystd. from MeOH yielded 26% dimorphic 2-acetoxy-6,7-benzo-4-carbomethoxy-2-methyl-9-oxobicyclo[1.3.3]nonane, m. 126.5°, 129.0-30.0°, λ 5.85 μ , n.m.r. peaks at 167, 177, 193 cycles/sec., consistent with assigned structure. The model reaction of IV with OHCCO₂Et was not reproduced with II. Na (0.10 g.) in 40 ml. absolute alc., 3.0 g. IV, and 4.5 g. EtOCH(OH)CO₂Et, b. 134.5-6.0°, refluxed 16 hrs. under a Dean-Stark head, the mixture acidified with dilute HCl, extracted with Et₂O, and the wine-red oil (4.05 g.) chromatographed on 100 g. Florisil with ether-C₆H₆Et₂O eluent sequence

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 1:9 Me₂CO-petr. ether, and the fractions recrystd. from MeOH yielded 39.0% XIII, m. 148.2-8.7°, λ 5.80 μ . Xylene (500 ml., distd. from Na) refluxed with distn. (N atm.) to remove the last traces of H₂O, 50 ml. transferred to a Hershberg dropping funnel, 548 mg. pulverized XIII (dried 48 hrs. at 25°/0.1 mm.), the 450 ml. xylene stirred with a Vibro-Mixer with addn. of 250 mg. freshly cut Na, the XIII in xylene added dropwise in 2 hrs., the refluxing and stirring continued 2 hrs., the cooled soln. treated with 10 ml. AcOH and a large vol. of H₂O, the dried and filtered xylene distd. (N atm.) in vacuo, and the residue (330 mg.) chromatographed on 10 g. Al₂O₃ yielded 17% material, m. 100-40°, on elution with 1:19 Me₂CO-petr. ether, recrystd. from (Me₂CH)₂O to give 4b,5,6,7,9,10-hexahydro-7-oxo-2,3,4-trimethoxy-A δ -B-homophenanthrene, m. 140.1-40.6°, λ 233 μ (e 21,600), λ 6.05, 6.18 μ . Further elution with 1:4 Me₂CO-petr. ether gave 80 mg. unsatd. cyclic β -oxo ester, m. 90-117°, λ 218, 252 μ (e 37,000, 15,000), λ 2.9-4.0°, 5.8-5.9 μ , tentatively regarded as the direct Dieckmann product. XI (4.394 g.) in 60 ml. MeOH refluxed 21 hrs. with 25 g. KOH in 100 ml. H₂O, the mixt. worked up as for XIV except that the solid diacid pptd. from the aq. soln. on acidification, the ppt. combined with the residue from CHCl₃ extn., and recrystd. from MeOH yielded 91.8% XV, m. 206.0-8.0° (MeOH); di-Me ester (XVI) m. 84.2-5.4°, λ 2.96, 5.81 μ . XVI (217 mg.) and 5 mg. p-MeC₆H₄SO₃H₂O heated 19 hrs. at 95° in 5 ml. AcOH, the product chromatographed on 10 g. Al₂O₃, and eluted with 1:49 Me₂CO-petr. ether yielded 32.2% material, m. 97-100°, recrystd. to give pure Me 6-acetoxy-6-carbomethoxymethyl-2,3,4-trimethoxybenzocycloheptane-5-propionate, m. 99.0-9.8°, λ 5.8 μ . XV (3.620 g.) in 22 ml. dry C₅H₅N treated with 2.025 g. purified C₆H₁₁N: C: NC₆H₁₁, the mixt. kept 20 hrs. at 25°, freed from 1.921 g. (C₆H₁₁NH)₂CO, m. 230-5°, the lactone acid residue (5.1 g.) taken up in MeOH, treated with freshly distd. CH₂N₂-Et₂O, the residue on evapn. chromatographed on 120 g. Al₂O₃, eluted with 1:9-1:4 Me₂CO-petr. ether, and recrystd. from MeOH gave 1.458 g. product, m. 147.5-50.0°, recrystd. to give 6-carbomethoxymethyl-6-hydroxy-2,3,4-trimethoxybenzocycloheptane-5-propionic acid δ -lactone (XVII), m. 149.8-50.3°, λ 5.80 μ . XVI (733 mg.) was pulverized and dried 24 hrs. at 25°/0.1 mm., added quickly to 260 ml. anhyd. Et₂O, and kept 6 days in a sealed dropping funnel. Anhyd. Et₂O (100 ml.) and 150 ml. liquid NH₃ vigorously stirred (N atm.) with 180 ml. freshly cut Na in a flame-dried app., treated dropwise with XVI in Et₂O, the NH₃ evapd., the residue dild. with 3 ml. MeOH in 50 ml. Et₂O, the mixt. acidified with 50 ml. 5% HCl, the aq. layer extd. with CHCl₃, the org. exts. washed with 5% aq. NaHCO₃, the washed and dried exts. evapd., the residue (566 mg.) chromatographed on 20 g. Al₂O₃, eluted with Me₂CO-petr. ether to remove 12.5 g. material, m. 165-76°, λ 2.97, 5.90 μ , consuming 84 equiv. HIO₄, and eluted with 1:1 Me₂CO-petr. ether yielded 9.2% cyclic hemiketal (XVIII), m. 149-54°, λ 282 μ (e 1200), λ 3.0, 5.82 μ . Further elution with 1:3 MeOH:Me₂CO gave 7 mg. material, m. 207-21°, λ 3.05, 5.7, 5.85 μ , consuming 1 mol. HIO₄. XVIII (62.7 mg.) refluxed 3 hrs. with 750 mg. Cu(OAc)₂ in 15 ml. MeOH, the mixt. dild. with 90 ml. N HCl, extd. with CHCl₃, the exts. washed with N HCl, dild. aq. NaHCO₃, and 25 ml. H₂O, the dried filtered ext. evapd., and the residue triturated with Et₂O gave 65 mg. material, m. 192-201°, λ 5.68 μ , repeatedly recrystd. from Me₂CO-petr. ether to give 7a-hydroxy-10-oxodecathoxydeacetamidohexahydrocolchicine (XVIII), m. 203.0-4.5° (decomp.). XVIII (50.3 mg.) and 50 mg. p-MeC₆H₄SO₃H₂O refluxed 5.5 hrs. in 100 ml. distd. C₆H₆ under a Dean-Stark head with development of a peak at 371 μ (e 13,000), extd. with dil. NaHCO₃ and H₂O, the

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 washings extd. with CHCl₃, the combined dried org. layers evapd. (N atm.), the residue [50 mg., λ 365 m μ (ϵ 9000, 95% alc.)] taken up in 50 ml. CHCl₃, treated with 25.0 mg. (CH₂CO)ZnBr, refluxed 15 min., the washed and dried soln. (λ 351 m μ , ϵ 7500, CHCl₃) evapd. (N atm.), the residue (53 mg.) chromatographed on 1.0 g. silicic acid, eluted with 1:1 CHCl₃-CCl₄, and the fraction, m. 150-65°, recrystd. from abs. alc. gave 3 mg. III, m. 165-7°, λ 244, 351 m μ (ϵ 35,600, 18,400). λ 6.19-6.25, 6.46 μ , identical with material obtained from I. The intensity at 351 m μ indicated 40% conversion of XVIII to III. III (5.00 g.) in 200 ml. MeOH methylated with CH₂N₂ in Et₂O, the solvents evapd., the oily residue dild. with 350 ml. Et₂O, and kept overnight gave 2.10 g. material, m. 179-84°, recrystd. from Me₂CO-petr. ether to give deacetamidocolchicine (XIX), m. 183.6-4.1°. The mother liquors evapd., and the residue fractionated from Et₂O-(Me₂CH)₂O yielded 24.3% deacetamidoisocolchicine (XX), m. 148.0-8.7°. The entire mother liquors from XIX and XX, as well as most of XIX, were reconverted to III by aq. acid hydrolysis to give material for remethylation to XX. XX (592 mg.) in 35 ml. hot CCl₄ stirred magnetically with 308 mg. recrystd. anhyd. (CH₂CO)ZnBr under reflux with ultraviolet light irradiation 23 min., the mixt. kept 1 hr. before filtration from pptd. (CH₂CO)ZnH, the filtrate partitioned between CHCl₃, the aq. layer washed with CHCl₃, the combined dried and filtered org. layers evapd., the oily product triturated with Me₂CO, and the cryst. produce (125 mg., m. 110-60°) repeatedly recrystd. from Me₂CO-petr. ether gave the bromo compd. (XXI), m. 161.0-3.5° (decompn.). Crystn. of the Me₂CO wash gave a nuclear bromination product, m. 218-22° (decompn.). XXI (56 mg., m. 163-8°) in 10 ml. MeOH and 34 mg. activated NaN₃ heated (H₂O bath) 27 hrs. at 90-5°, the cooled product partitioned in H₂O-CHCl₃, the dried and filtered CHCl₃ evapd., and the residue (53 mg.) recrystd. from Me₂CO-petr. ether yielded 65% pure 7-azidoacetamidocolchicine (XXII), m. 166-9°, λ 4.72 μ . XXII (21 mg.) in 10 ml. MeOH hydrogenated 10 min. at 25°/1 atm. with 21 mg. 10% Pd-C, filtered through Filtercel, evapd. (N atm.), the product (44 mg. from 2 runs) refluxed 2 hrs. in 10 ml. 1.0N HCl, adjusted to pH 7.5 with dil. aq. NaHCO₃, extd. with CHCl₃, the partially cryst. material (40 mg., m. 210-40°) chromatographed on 2.0 g. silicic acid, eluted with CHCl₃ and 1:49 MeOH-CHCl₃, and the fractions (16 mg.) recrystd. from abs. alc. yielded racemic trimethylcolchicine acid (XXIII), m. 240-4° (decompn.), λ 230(shoulder), 242, 353 m μ (ϵ 27,000, 31,000, 19,000), identical with a specimen prepd. according to Corradi and Hardegger (CA 52,343d). Conversion of III to XXIII marked the successful culmination of a series of steps constituting the total synthesis of I. The more plentiful hydroxycyano ester XII (11.255 g.) in 100 ml. MeOH refluxed 24 hrs. with 50 g. KOH in 200 ml. H₂O, the MeOH evapd., the basic soln. acidified to Congo red by dil. HCl, extd. with CHCl₃, the oily product triturated with Me₂CO-petr. ether, and recrystd. yielded 94.7% XIV, 6-carboxymethyl-6-hydroxy-2,3,4-trimethoxybenzocycloheptane-5-propionic acid, m. 80-90°; di-Me ester m. 100.6-1.4° (petr. ether), λ 2.90, 5.85 μ . XXIII (2.096 g.) and 10 ml. MeI in 40 ml. purified dioxane heated at 74° (previously heated oil bath) 24 hrs., the cooled mixt. kept 16 hrs., and the ppt. washed with purified dioxane gave 1.551 g. material, m. 200-20°, crystd. from 40 ml. abs. alc. to yield 31.6% N,N-dimethyldeacetylcolchicine hydroiodide, m. 250-2° (decompn.). The salt (0.964 g.) shaken thoroughly with 100 ml. 1:1 CHCl₃-H₂O, the aq. layer extd. with CHCl₃, the dried and filtered exts. evapd. in vacuo, the residue heated 42 hrs. at 74° in 50 ml. purified dioxane, the dioxane-washed product dried in air, and a sample of the material (0.865

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 AB The effect was studied of Ba⁺⁺, Pb⁺⁺, Mg⁺⁺, Al⁺⁺⁺, Cr⁺⁺⁺, Mn⁺⁺, Fe⁺⁺⁺, Co⁺⁺, Ni⁺⁺, Zn⁺⁺, Cd⁺⁺, Cu⁺⁺, Ag⁺, and K⁺, at different H⁺ concns., on oxidation of sulfite. The same cations can have an opposite effect depending on the pH. The strongest inhibitors were Cu⁺⁺ (in an acid medium) and Cr⁺⁺⁺ (in an alkaline medium). The strongest promoters were Fe⁺⁺⁺, Co⁺⁺, Ni⁺⁺, and Cu⁺⁺ in an alkaline medium and Ag⁺ and Mn⁺⁺ in an acid medium. With the simultaneous introduction in the solution of 2 cations having the same effect a mutual intensification of their effect did not occur. When two cations having an opposite influence were added, one suppressed the effect of the other.
 ACCESSION NUMBER: 1962:35422 CAPLUS
 DOCUMENT NUMBER: 56:35422
 ORIGINAL REFERENCE NO.: 56:6696a-c
 TITLE: Catalytic oxidation of a sulfite solution
 AUTHOR(S): Dmitriev, A. K.; Kustodina, V. A.
 SOURCE: Trudy Leningrad. Tekhnol. Inst. (1959), (No. 7), 175-7
 From Referat. Zhur., Khim. 1961, Abstr. No. 9B436.
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 g. m. 231.5-5.5°) recrystd. from abs. alc. and dried at 25°/0.1 mm. 18 hrs. gave N,N,N-trimethylcolchicine acid iodide, m. 231.8-5.4° (decompn.), λ 219, 244, 358 m μ (ϵ alc.). The quaternary ammonium iodide (4.785 g.) in 130 ml. 0.385N NaOH hydrogenated at 25°/1 atm. with 1.0 g. 10% pre-equilibrated Pd-C 1 hr., the filtered soln. acidified with Ac₂O, extd. with CHCl₃, the dried filtered ext. evapd. in vacuo, the slightly oily solid crystd. from abs. alc., and the 3 crops (2.095 g. m. 168-70.5°, 0.168 g. m. 167.5-8.5°, 0.065 g. m. 167.8-9.0°) combined yielded 76.7% III, recrystd. from abs. alc. to give golden prisms, m. 169.3-70.5°, λ 245, 351 m μ ($\log \epsilon$ 4.55, 4.26, alc.).
 ACCESSION NUMBER: 1962:46205 CAPLUS
 DOCUMENT NUMBER: 56:46205
 ORIGINAL REFERENCE NO.: 56:8779b-i, 8780a-i, 8781a-i, 8782a-g
 TITLE: Synthesis of colchicine
 AUTHOR(S): Tamelen, E. E. van; Spencer, T. A. Jr.; Allen, D. S. Jr.; Orvis, R. L.
 CORPORATE SOURCE: Univ. of Wisconsin, Madison
 SOURCE: Tetrahedron (1961), 14, 8-34
 CODEN: TETRAH; ISSN: 0040-4020
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 AB When AuCl₃ was added to an ether solution of diazomethane, colloidal Au was formed and became surrounded by polymethylene. Metallic Au was assumed to be the catalyst, and it was found that when an ether solution of diazomethane was put in contact with a Au film formed by evaporation of the metal under high vacuum, the diazomethane decomposed to yield a polymer in nearly quant. yield. Similarly, diazoethane yielded crystalline polyethylidene, the Au surface exhibiting a stereoregulating activity. Evaporated films of the following metals, in decreasing order of yield, gave amorphous polymers from ether solns. of diazoethane: Cu, Ti, Fe, Mg, W, Ni, Mn, V, Ta, Pt, Co, Zn, Cd, Au (1% yield of crystalline polyethylidene), Cr, Al while Mo, Pd, Rh, Zr, and Ag gave no polymer, although they did cause decomposition of the diazoethane. Apparently crystalline polymers from ether solns. of diazopropane and diazobutane were obtained when AuCl₃ was used as a catalyst. The yields were
 51-2%.
 ACCESSION NUMBER: 1962:25712 CAPLUS
 DOCUMENT NUMBER: 56:25712
 ORIGINAL REFERENCE NO.: 56:4944a-c
 TITLE: Reactions of diazo alkanes upon metallic surfaces: polymer formation and a stereoregulating action of gold
 AUTHOR(S): Nasini, A.G.; Trossarelli, L.; Saini, G.
 CORPORATE SOURCE: Univ. Turin, Italy
 SOURCE: Makromolekulare Chemie (1961), 44-46, 550-69
 CODEN: MACEAK; ISSN: 0025-116X
 DOCUMENT TYPE: Journal
 LANGUAGE: English

L20 ANSWER 190 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB The H exchange between isovaleraldehyde (I) and MeOH, EtOH, or iso-PrOH in gaseous phase over Cu, Ni, ZnS, ZnO, MgO, or CdO on pumice was examined. The temperature dependence of the yield of isocamyl alc. (II) at constant flow rate and constant mol. ratio of I to MeOH, EtOH, or iso-PrOH (1:1.89) was studied in the range 300-500°. The yields of II (and optimum temps. in brackets) for the I-MeOH, I-EtOH, and I-iso-PrOH systems, resp. are: Cu 0, 32 (308°), 28 (380°); Ni 0, 55 (230°), 8.5 (320°); ZnS 0, 45 (360°), 53 (375°); ZnO 0, 44.5 (400°), 35.5 (450°); MgO 21.1 (460°), 58.6 (375°), 52 (450°); CdO on pumice 0, 21 (375°), and 17.21 (425°). Thus, the most efficient catalyst is MgO. The course of H exchange is nearest to the theoretical equilibrium in case of I-EtOH mixts. Cf. CA 55, 10300c.
ACCESSION NUMBER: 1962:5247 CAPLUS
DOCUMENT NUMBER: 56:5247
ORIGINAL REFERENCE NO.: 56:9731,974d-e
TITLE: Hydrogen exchange in the gaseous phase. III
AUTHOR(S): Malinowski, Stanislaw; Kobylinski, Tadeusz
CORPORATE SOURCE: Politech., Warsaw
SOURCE: Roczniki Chemii (1961), 35, 917-30
CODEN: ROCHAC; ISSN: 0035-7677
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L20 ANSWER 191 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
AB It is not possible to determine very small amts. of Zn polarographically in the presence of large amts. of Ni. Zn must be removed previously as ZnS. The polarographic determination of Zn in concns. <0.5% in 5% (NH₄)₂C₂O₄, in 0.5M K.H tartrate, or in alkaline tartrate solution yielded results which agreed within 3%. The catalyst samples were dissolved directly in the oxalate or tartrate solution. They were difficultly soluble in HCl. Pb, Cu, and Fe did not interfere.
ACCESSION NUMBER: 1962:164 CAPLUS
DOCUMENT NUMBER: 56:164
ORIGINAL REFERENCE NO.: 56:22c-d
TITLE: Polarographic determination of zinc in nickel catalysts
AUTHOR(S): Witwicka, Jadwiga
SOURCE: Przemysl Spozyczy (1958), 12, 157-8
From: C.Z. 1959, 12953.
CODEN: PRSPAD; ISSN: 0033-250X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB Six new halogen-containing disubstituted phenothiazines, RR'C₁₂H₇N₃ (I), were synthesized by iodine-catalyzed thionation of appropriate diphenylamines, RCGH₄NHCGH₄R' (II), prepared by the Weston and Adkins modification of the Goldberg reaction (CA 22, 1336). Anhydrous K₂CO₃ (8.4 g.), 16.5 g. 3-MeOCGH₄NHAc, 33 g. 3-BrCGH₄CF₃, and 0.34 g. Cu bronze powder stirred 24 hrs. under reflux at 240° (bath temperature), the cooled mass extracted with Me₂CO, the residue on evaporation refluxed 4 hrs. in 100 ml. 20% alc. KOH, poured into 200 ml. saturated NaCl solution, and extracted with Et₂O yielded 72% oily II (R = 3-MeO, R' = CF₃) (III), b.p. 148-8.5°, n_D20 1.5630. Similarly were prepared II [compound, R, R', m.p. (petr. ether), b.p./mm., n_D/temperature, and % yield given]: (IV), 3,4-Cl(F), 4-F, 56.5-57°, 145-50°/1.0. -, 30; (V), 3-CF₃, 4-Me, 66-7°, -, -, 54; (VI), 3-F, 3-CF₃, -, 135-5.5°/2, 1.5521/21°, 62. VI (7.65 g.), 1.73 g. S, and 0.23 g. iodine maintained 2.5 hrs. at 150°, the mass taken up in 100 ml. boiling C₆H₆, treated with decolorizing C, and the filtered solution concentrated in vacuo yielded 12% I (R = 2-F, R' = 8-CF₃) (VII), m. 159.5-60°, producing a deep red color with concentrated HNO₃. Thionation could not be initiated at 250° with 1% iodine. VI (0.01 mole), 0.018 mole S, and 0.13 g. iodine maintained at 135-40° 1.5 hrs., the mass taken up in boiling C₆H₆, the solution treated with Norit, concentrated in vacuo, and the tan solid (0.6 g.) sublimed at 120°/0.05 mm. yielded 0.1 g. yellow crystals, m. 149-51°, an addition compound of VII + 2 S atoms. V (75 g.), 17.3 g. S, and 0.75 g. iodine kept 1.5 hrs. at 150-60° and 30 min. at 170°, the mixture extracted with C₆H₆, and the extract concentrated yielded 18% I (R = 3-Me, R' = 8-CF₃) (VIII), m. 216-17° (C₆H₆). In a similar thionation, 2.51 g. V, 0.58 g. S, and 25 mg. iodine raised from 150 to 230° during 3 hrs., maintained at 230° 15 min., the mixture recrystd. from CCl₄ (Norit), and sublimed in vacuo gave pale yellow crystals, m. 202-3°, an addition compound of VIII + 6 S atoms. III (8.0 g.), 1.73 g. S, and 0.24 g. iodine kept 1 hr. at 130-40° and 1 hr. at 140-55°, the mixture taken up in 100 ml. boiling C₆H₆, decolorized by stirring with activated Al₂O₃, concentrated in vacuo, and the platelets, m. 168.0-8.5°, recrystd. from petr. ether gave a methoxy(trifluoromethyl)phenothiazine in combination with 4 S atoms, m. 173-3.5°, recrystd. from CCl₄ to give a pure methoxy(trifluoromethyl)phenothiazine (IX), m. 179-80°. III (93.5 g.), 22.4 g. S, and 1.4 g. iodine kept 2 hrs. at 175-80°, the mixture taken up in CCl₄, the decolorized solution (Al₂O₃) concentrated, the yellow plates (m. 136-8°) sublimed in vacuo, and then recrystd. from C₆H₆ yielded 18% methoxy(trifluoromethyl)phenothiazine (X), m. 137.5-9.0°. IX and X had almost identical spectra with strong peaks at 12.3 and 12.7 μ, but no definite structures could be presently assigned. An attempt to synthesize the desired I (R = 2-MeO, R' = 8-CF₃) (XI) via an unambiguous Smiles synthesis was unsuccessful. Alc. (55 ml.) containing 5.2 g. [2,4-H₂N(CF₃)CGH₃] 2% in stirred (N atmospheric) with 0.023 mole NaOEt in 25 ml. alc. at 20°, treated with 4.29 g. 3,4-Cl(O₂N)CGH₃OME in 55 ml. alc., the mixture refluxed 16 hrs., the filtered solution concentrated in vacuo, and the residue recrystd. from alc.-H₂O yielded 38% material, sublimed in vacuo and recrystd. from Et₂O-petr. ether to give 2-amino-5-methoxy-2'-nitro-4-

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(trifluoromethyl)diphenyl sulfide (XII), m. 139.5-41°. XII (3 g.) and 30 g. 90% HCO₂H refluxed overnight, poured onto ice, and the brown solid recrystd. from MeOH yielded 4% 2-formamido-5'-methoxy-2'-nitro-4-(trifluoromethyl)diphenyl sulfide (XIII), m. 188.5-90°. XIII (0.9 g.) in 10 ml. Me₂CO refluxed 75 min. with 5.2 ml. N alc. NaOH, the filtered soln. concd. in vacuo, the residue taken up in alc., acidified with dil. HCl, filtered, and the yellow crystals recrystd. from alc. gave II (R = 2,5(O₂N)(OMe), R' = CF₃) (XIV), m. 114-15°. The filtrate exposed 16 hrs. to the air at room temp. turned green and gave orange crystals of the disulfide, C₂₈H₂₀F₆N₄O₆S₂, m. 163-4° (alc.). It was not found possible to prep. 2-chloro-3,7-difluorophenothiazine by treating 3-chloro-4,4'-difluorodiphenylamine with S and iodine, since thionation was invariably accompanied by loss of an F atom. Elimination of catalyst or substitution of AlCl₃ for iodine were of no avail. Abs. alc. (1 l.) contg. 153.6 g. 2,3-Cl₂CGH₃NO₂ stirred 5 min. at 20° with 32 g. NaOH and 100 g. o-H₂NCGH₄SH in 2040 ml. 2:15 H₂O-abs. alc., the mixt. refluxed 2 hrs. with stirring, and the hot filtrate boiled with addn. of 800 ml. H₂O with stirring gave 197 g. 2'-amino-6-chloro-2-nitrodiphenyl sulfide, m. 126-7°, converted by refluxing 10 hrs. with 2 kg. HCO₂H, pouring over 2 l. crushed ice, and recrystg. from dil. alc. (Norit) to 142 g. 6-chloro-2'-formamido-2-nitrodiphenyl sulfide (XIV), m. 124-5°. XIV (6.2 g.) in 75 ml. Me₂CO refluxed 2 hrs. with 20 ml. N alc. NaOH, the filtered soln. evapd., and the product recrystd. from alc. gave 2.4 g. 1-chloro-10-formylphenothiazine, m. 112-13°, green with HNO₃. XIV (62 g.) in 750 ml. Me₂CO refluxed 2.5 hrs. with 400 ml. N alc. NaOH, the filtrate evapd., and the product recrystd. from CCl₄ and alc. yielded 43% 1-chlorophenothiazine, m. 92-3°, λ 12.8, 13.1, 13.4 μ, no peaks at 12.0-12.5 μ. The infrared spectra of some disubstituted phenothiazines between 11.0 and 13.5 μ were tabulated and used for assignment of tentative structures of VII and VIII.
ACCESSION NUMBER: 1961:124886 CAPLUS
DOCUMENT NUMBER: 55:124886
ORIGINAL REFERENCE NO.: 55:23543d-1,23544a-f
TITLE: Synthesis of phenothiazines. V. Halogen-containing phenothiazines
AUTHOR(S): Nodiff, Edward A.; Craig, Paul N.
CORPORATE SOURCE: Temple Univ., Philadelphia, PA
SOURCE: Journal of Organic Chemistry (1961), 26, 824-8
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 55:124886

L20 ANSWER 193 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB A process was described for reductively alkylating an aromatic and amino compound with a ketone in the presence of H₂ and subsequent dehydrogenating the alc. into a ketone. Thus, by using a 1:8 mole ratio, p-O₂N₂C₆H₄NH₂ (I) was alkylated with MeCOEt (II) over a 10:10:1 Cr₂O₃-CuO-BaO catalyst at 160° and a H₂ pressure of 60 atmospheric. The N,N'-di-sec-butyl-p-phenylenediamine was separated from H₂, unreacted II, H₂O, and sec-alc. amino compound (III). While H and II were recycled to the alkylator, III was fractionated to sep. sec-BuOH (IV) and amino compound IV was dehydrogenated over a catalyst (60% Cu, 37% Zn, and 3% Pb). While II and H were separated from unreacted IV and recycled to the alkylator, unreacted IV was recycled to the dehydrogenator. The products were especially useful as antioxidants for motor fuels.

ACCESSION NUMBER: 1961:87325 CAPLUS
 DOCUMENT NUMBER: 55:87325
 ORIGINAL REFERENCE NO.: 55:16480g-i
 TITLE: Combined alkylation and dehydrogenation process, especially for production of N,N-di-sec-alkyl-p-phenylenediamines
 INVENTOR(S): Chemicke, Joseph A.
 PATENT ASSIGNEE(S): Universal Oil Products Co.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2969394		19610124	US	

L20 ANSWER 194 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The reaction of tolylene diisocyanate with a secondary hydroxyl-containing polyoxypropylene triol was studied in the presence of numerous compds. to test their catalytic effect on the gelation time of the reaction mixture Compds. in roughly descending catalytic activity are: Bi, Pb, Sn, triethylenediamine, strong bases, Ti, Fe, Sb, U, Cd, Co, Th, Al, Hg, Zn, Ni, trialkyl amines, Ce, Mo, V, Cu, Mn, Zr, trialkyl phosphines. Similar gelation tests were made with m-xylylene diisocyanate and with hexamethylene diisocyanate and the triol. In these tests the decreasing order of strength of the catalysts was found to be Bi, Fe, Sn, Pb, Ti, Sb, strong bases, Co, Zn, triethylenediamine, trialkyl amines. A mechanism is proposed for the way in which a metal compound may catalyze the isocyanate-hydroxyl reaction. Steric hindrance is suggested to explain the differing catalytic activities observed with tolylene diisocyanate and the unhindered aliphatic diisocyanates.

ACCESSION NUMBER: 1961:46778 CAPLUS
 DOCUMENT NUMBER: 55:46778
 ORIGINAL REFERENCE NO.: 55:9010f-h
 TITLE: Catalysis of the isocyanate-hydroxyl reaction
 AUTHOR(S): Britain, J. W.; Gemeinhardt, P. G.
 CORPORATE SOURCE: Mobay Chem. Co., New Martinsville, WV
 SOURCE: Journal of Applied Polymer Science (1960), 4, 207-11
 CODEN: JAPNAB; ISSN: 0021-8995
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 195 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Foams with a d. of <5 kg./cu. m. are prepared from liquid aminoplasts soluble in H₂O to yield 50-90% solns. having viscosities of 1000-6000 cp. The aminoplasts may be urea-guanidine carbonate-HCHO, or melamine-guanidine carbonate-HCHO, or based on aniline and a guanidine derivative. Furfural, AcH, hexamethylenetetramine, or mixts. of these with HCHO may be used. As blowing agent, a peroxide of Ca, Ba, Zn, Na, Pb, Mn, or Mg is used. Plasticizers include polyhydric alcs., such as glycerol, mannitol, and sorbitol, liquid resins based on vinyls, cellulosic matter, rosin, or casein, or synthetic materials, such as tritolyl phosphate, sebacates, or adipates. An emulsifier, such as soap, Na stearate, Zn stearate, oleate, and palmitate, or synthetic emulsifiers, based on polyethylene glycol or sulfonates and a catalyst, such as NH₄Cl or mineral acids, are added. The resin is made in aqueous medium and adjusted to the correct concentration and viscosity. The peroxide, surface-active agent, catalyst, and plasticizer are added in that order, and the mixture is poured into cellophane-protected or oil-coated molds heated to 30-300° depending upon the type and concentration of peroxide and other ingredients. The products are not friable, noninflammable, of more or less closed cell structure, impermeable to H₂O, and resistant to strong acids.

ACCESSION NUMBER: 1960:122699 CAPLUS
 DOCUMENT NUMBER: 54:122699
 ORIGINAL REFERENCE NO.: 54:23425e-h
 TITLE: Cellular aminoplasts
 INVENTOR(S): Meyer, Robert
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1186572		19590827	FR	

L20 ANSWER 196 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB A gas-phase continuous process is given for the manufacture of aromatic N-alkylamines (along with small amts. of primary amines) from aromatic nitro compds., alcs., and H₂ in the presence of a catalyst promoting hydrogenation and dehydration. In an example, the catalyst is prepared by saturating an Al₂O₃-kaolin carrier with Cu(NO₃)₂, alone or mixed Zn(NO₃)₂, drying, heating in O₂, and activating with H₂. A 1:1.5 mole PhNO₂-EtOH mixture, containing the circulating H₂ in excess, is passed through this catalyst at 270° and a space velocity of 300 vols./volume/hr. to yield 20% PhNH₂, 65% PhNHET, and 15% PhNET₂. Similarly, when the ratio is 1:0.4 mole, 65% PhNH₂, 20% PhNHET, and 15% PhNET₂ are obtained.

ACCESSION NUMBER: 1960:117984 CAPLUS
 DOCUMENT NUMBER: 54:117984
 ORIGINAL REFERENCE NO.: 54:22495h-1,22496a
 TITLE: Aromatic N-alkylamines
 INVENTOR(S): Kurowski, Stanislaw; Leszczynski, Zbigniew
 PATENT ASSIGNEE(S): Instytut Chemii Ogólnej
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PL 41483		19580710	PL	

L20 ANSWER 197 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The reduction of nitrostyrenes, nitrostilbenes, and nitrocinnamic acids with either Zn and NH₄Cl or Al-Hg in moist Et₂O gave the corresponding amines. Intermediate hydroxylamines were not isolable. o-(I) and p-O₂NC₆H₄CH₂CH₂ (II) were prepared by dehydrobromination of the corresponding bromides and the m-isomer (III) by decarboxylation of m-O₂NC₆H₄CH₂CHCO₂H (III) with Cu powder and quinoline. cis-o-O₂NC₆H₄CH₂CHPh (IV) was prepared by decarboxylation of trans-o-O₂NC₆H₄CH₂CHPhCO₂H with Adkins catalyst; trans-m-O₂NC₆H₄CH₂CHPh (V) by the Meerwein reaction; trans-p-O₂NC₆H₄CH₂CHPh (VI) by condensation of BzH and p-O₂NC₆H₄CH₂CO₂H; trans-(p-O₂NC₆H₄CH₂)₂ (VII) by treating p-O₂NC₆H₄CH₂Cl with KOH; o-O₂NC₆H₄CH₂CHCO₂H (VIII) by condensation of o-O₂NC₆H₄CHO with Ac₂O; III by condensation of m-O₂NC₆H₄CHO and CH₂(CO₂H)₂ in the presence of CSH₅N. To 10.0 g. Al-Hg and 400 ml. Et₂O in a 1 l. 2-necked flask equipped with a reflux condenser and a dropping funnel was added portionwise through the condenser 12.0 g. I, II, or III with occasional shaking while simultaneously adding slowly 8 ml. H₂O from the dropping funnel (a vigorous reaction occurred and addition of I, II, or III was regulated to give gentle reflux), the mixture kept until the reaction subsided, refluxed 10 min. on a H₂O bath, the precipitate filtered off, washed several times with Et₂O, the combined filtrate and washings dried, and evaporated 1st on a H₂O bath and then in vacuo gave (from I) 9.0 g. o-H₂NC₆H₄CH₂CH₂ (IX) (after distillation from 0.5 g. p-CH₃/(OH)₂ (X), 5.1 g. IX, b₁₀ 93-6°, n_D20 1.6065; (from IIa) 6.2 g. m-H₂NC₆H₄CH₂CH₂, b₆ 82-5° (distilled from X), n_D20 1.6069; and (from II) 8.6 g. p-H₂NC₆H₄CH₂CH₂ (XI) (after distillation from X, 5.8 g. XI, b₇ 74-7°, n_D20 1.6185; Ac derivative (XII) m. 135-6°). m-O₂NC₆H₄NH₂ reduced with Al-Hg in moist Et₂O as above, the mixture filtered, the filtrate dried, evaporated, and the residue distilled in vacuo gave 80% a-C₆H₄(NH₂)₂, m. 63°. NH₄Cl (5.0 g.) in 20 ml. H₂O added to 7.0 g. II in 75 ml. Me₂CO, the mixture heated to boiling on a H₂O bath, removed from the bath, treated portionwise with 10.0 g. Zn dust to maintain a moderate reaction, treated with an addnl. 5 g. Zn dust when the reaction subsided, refluxed 30 min. on a H₂O bath, filtered hot, the precipitate washed twice with Me₂CO, the combined filtrate and washings concentrated on a H₂O bath to 30 ml., and treated with Ac₂O and NaOAc gave 5.9 g. XII, m. 134°. IV, V, VI, VII, VIII, III, and p-O₂NC₆H₄CH₂CHCO₂H (XIII) were similarly reduced with Zn and NH₄Cl. In the reduction of cis-IV, the final Me₂CO solution evaporated on a H₂O bath until droplets of amine appeared on the surface, extracted with Et₂O, the extract dried, saturated with dry HCl, the precipitate filtered off, washed with Et₂O, and recrystd. from a small EtOH gave 72% o-H₂NC₆H₄CH₂CHPh.HCl (XIV), m. 199-201°. The reaction carried out in alc. gave 51% XIV. In the reduction of V and VI, the final Me₂CO solution treated with 15 ml. 3N aqueous NH₃ to dissolve the Zn(OH)₂, the solution concentrated on a H₂O bath to 30 ml., treated with sufficient H₂O containing a few drops concentrated aqueous NH₃ to precipitate the product, the precipitate filtered off, washed with 5% aqueous NH₃, and dried; in the case of V, the product was dissolved in Et₂O, the solution filtered, and the filtrate evaporated to give 77% m-H₂NC₆H₄CH₂CHPh (XV), m. 120-1°; in the case of VI, the crude product recrystd. from a little dilute EtOH gave 84% p-H₂NC₆H₄CH₂CHPh (XVI), m. 150-1°.

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 AB CF. CA 51, 12056c. A mixture of 45 g. 4-methoxy-2-nitrotoluene, 325 g. granulated Zn, and 400 cc. concentrated HCl was heated gently. Upon reacting 400 cc. HCl was added in small portions. After 3 hrs. the solution was cooled and alkalinized with aqueous NaOH to yield 4-methoxy-2-aminotoluene (I), needles, m. 47°, b. 253°. A solution of 13.7 g. I in 60 cc. 48% HBr was diazotized with 7.6 g. NaNO₂ in 20 cc. H₂O. The resultant compound was decomposed in a warm water bath, with Cu as a catalyst, to 4-methoxy-2-bromotoluene (II), b₂₃ 115°. II was then oxidized with Ac₂O-AcOH-concentrated H₂SO₄ to 4-methoxy-2-bromobenzaldehyde diacetate (III), m. 130-2°. A solution of III in concentrated HCl, water, and alc. was refluxed 1 hr. to give needles of 4-methoxy-2-bromobenzaldehyde (IV), m. 68-9°. Bromination of II with Br yielded IV directly. A mixture of 21.5 g. IV, 11.5 g. malonic acid, 25 cc. EtOH, and 25 cc. anhydrous pyridine was heated 8 hrs. on a water bath to yield needles of 4-methoxy-2-bromocinnamic acid (V), m. 250°. A mixture of 51.4 g. V, 130 g. Zn-Hg, 40 cc. glacial AcOH, and 240 cc. concentrated HCl was heated gently approx. 2 hrs. to form a precipitate of β-(4-methoxy-2-bromophenyl)propionic acid (VI), crystallized from EtOH to form needles, m. 116-18°. VI was then converted to the acid chloride and submitted to Friedel-Crafts acylation to obtain 6-methoxy-4-bromo-indan-1-one, yellow needles, m. 113-14°. Direct chlorination of 4-methoxy-2-cyanotoluene yielded 2 compds., 4-methoxy-2-cyano-3-chlorotoluene, needles, m. 106-7°, and 4-methoxy-2-cyano-3,5-dichlorotoluene, needles, m. 185-7°. The former was reduced with LiAlH₄ to form a compound, C₉H₁₀NOCl₂, m. 227-9°. The influence of substituents on the halogenation was shown by halogenating o-bromotoluene and p-methoxytoluene to o-bromobenzyl bromide and 4-methoxy-3-bromotoluene, resp., 18 references.
 ACCESSION NUMBER: 1960:97474 CAPLUS
 DOCUMENT NUMBER: 54:97474
 ORIGINAL REFERENCE NO.: 54:18452h-i, 18453a-c
 TITLE: Cyclopentenphenanthrene derivatives. V. Synthesis of 6-methoxy-4-bromoindan-1-one
 AUTHOR(S): Alberola, Angel; Lora-Tamayo, M.; Yahyaoui, Salah
 SOURCE: Anales real soc. espan. fis. y quim. (Madrid) (1959), 55B, 683-92
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 197 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN (Continued)
 AB The reaction carried out in EtOH gave 59% XV and 61% XVI. In the redn. of VII, the Me₂CO was removed, the residual mixt. dild. with H₂O, the ppt. filtered off, dried 30 min. at 100°, boiled with PhCl and 0.5 g. C, filtered while hot, the filtrate allowed to cool 6 hrs. in an ice bath, the ppt. filtered off, and dried to give 60% (H₂NC₆H₄CH₂)₂, m. 227-8°. In the redn. of VIII, III, and XIII, the reaction mixt. was filtered while hot, the ppt. extd. 3 times with 15 ml. warm 3N aq. NH₃, the Me₂CO filtrate concd. on a H₂O bath to 30 ml., and combined with the aq. NH₃ solns.; in the case of XIII, the soln. acidified with excess AcOH, the ppt. filtered off, washed once with cold dil. AcOH and once with ice-cold H₂O, and dried gave 74% crude p-H₂NC₆H₄CH₂CHCO₂H, m. 175-6° (decompn.) (H₂O); in the case of III, the soln. acidified with 30 ml. concd. HCl, cooled 3 hrs. in an ice bath, the ppt. filtered off, dissolved in boiling H₂O, the soln. treated with excess aq. NaOAc, the ppt. filtered off, washed with ice-cold H₂O, boiled with 50 ml. H₂O, the soln. filtered while hot, the filtrate cooled in an ice bath, and the ppt. collected (an addnl. amt. of product was obtained from the filtrate) gave 70% (total) m-H₂NC₆H₄CH₂CHCO₂H, m. 181°; in the case of VIII, the soln. acidified, concd. to half its vol., satd. with HCl, stored overnight at 0-5°, the ppt. collected, dissolved in boiling H₂O, reprecipd. with NaOAc, washed with ice-cold H₂O, dried, and recrystd. from PhMe gave 63% o-H₂NC₆H₄CH₂CHCO₂H, m. 157-8°. Attempts to repeat Alway and Bonner's (Amer. Chem. J. 32, 392 (1904)) prepn. of m- and p-ONC₆H₄CH₂CHCO₂H were unsuccessful, since in each case redn. of nitrocinnamic acids afforded amines and not hydroxylamines.
 ACCESSION NUMBER: 1960:117970 CAPLUS
 DOCUMENT NUMBER: 54:117970
 ORIGINAL REFERENCE NO.: 54:22491a-i, 22492a-b
 TITLE: Reduction of vinyl aromatic nitro derivatives
 AUTHOR(S): Boyer, J. H.; Alul, H.
 CORPORATE SOURCE: Tulane Univ., New Orleans, LA
 SOURCE: Journal of the American Chemical Society (1959), 81, 2136-7
 CODEN: JACSAT; ISSN: 0002-7863
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 54:117970

L20 ANSWER 199 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB The process of continuous hydrogenation under pressure was carried out in an apparatus in which H was introduced into a receiver working under 0.5-atmospheric excess pressure; upon increasing the pressure above a determined value, the feeding line closed automatically, and when the pressure fell to 0.1 atmospheric, the compressor also stopped automatically, forcing H into 2 buffers at 400 atmospheric; one of the buffers served to feed H to the continuously working device mounted separately; the substance to be hydrogenated was forced into the mixing 3-way pipe by means of a high-pressure pump and H was introduced from the buffer; the mixture was directed into 2 0.5-l. tubes filled with suitable catalysts and fitted with a 3-zone elec. furnace (manometers and heat gages were installed at different points); the product, after passing through the reactor, was cooled in a condenser and collected in a receiver-separator out of which H entered the atmospheric through a throttle valve and a gas counter; the hydrogenation product also passed through a throttle valve into a collector at atmospheric pressure. A continuous process for obtaining furyl alc. (I) was developed by using the above apparatus in which Cu chromite, stabilized with alkaline earth metal oxides, was used as a catalyst. This catalyst was also found to be most suitable for the hydrogenation of carbonyl groups or in similar cases, e.g., the hydrogenation of hydroxyvaleric aldehyde in pentanediol (in this case, by a batch process). The hydrogenation of furan (II) to yield tetrahydrofuran (III) was carried out by introducing it together with H in the tubular reactor filled with skeletal Ni; heating was accomplished by circulating a liquid heated to constant temperature (aqueous ethylene glycol (IV) with a constant b.p.). The continuous process of hydrogenation of nitriles into amines (e.g. the dinitrile of adipic acid) was carried out to give 85% basic products on skeletal Co, in MeOH saturated with NH₄OH. The yield of nitrites prepared from chlorides by the action of alkali metal cyanides was increased by working at atmospheric pressure, but by using high-boiling solvents, e.g., aqueous glycol for the preparation of dinitriles from dichlorobutane (V) and dichlorodibutyl ether (VI), adiponitrile in the preparation of chlorovaleronitrile, and glycerol in the synthesis of the nitrile of hydroxycaproic acid. In all cases the yield was remarkably increased. The esterification of chlorides for obtaining the complex esters required in the plastic industry was used successfully with salts of fatty acids. Furfural (VII) obtained from the peat industry was quite unsuitable for the synthesis of "semi-products." VII obtained from the hydrolysis of resinous wood was not used either, owing to the presence (even in small quantities) of compds. of the terpene series which cause the formation of resins. The hydrogenation of VII into tetrahydrofurfuryl alc. (VIII) was carried out in 2 stages, and satisfactory results were obtained at 95-100°, under a pressure of 100 atmospheric, and a volume rate of 0.12-0.3, during 350 hrs.; under these conditions the moist product contained 97-98% I and the content of VII did not exceed 0.2%. I was then converted into VIII (yield 78%) by the batch process at 130-5°, under a pressure of 100 atmospheric with Ni on Cr oxide as the catalyst, or by the continuous process at 120-5°, 100 atmospheric, with Ni on Cr oxide, and a volume rate of 0.2. The crude hydride was obtained in a 100% yield (on the weight of I) and contained 90% VIII and 0.2-0.3% I. VIII, b. 177-8°, d. 1.050, n_D 1.4502, was mostly used in further syntheses: VIII with SOCl₂ in the presence of CSH₅N yielded 75%

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 tetrahydrofurfuryl chloride (IX), b₇-8 37-8°, d₂₀ 1.1112, n_D 1.4556. IX with NaH₂ in liquid NH₃ yielded 65% 4-pentyn-1-ol (X), b₇ 47°, d₂₀ 0.9132, n_D 1.4455, hydroxyl no. 19.7. X in the presence of CuCl and NH₄Cl was oxidized in an aq. soln. of O of the air into 95% 4,6-decadiene-1,10-diol which in its turn, with Raney Ni catalyst at room temp. and atm. pressure yielded 1,10-decanediol in a quant. yield; the oxidation of this diol with HNO₃ yielded 90% sebacic acid. The dehydration and the simultaneous isomerization of VIII carried out at 340-60° over activated Al₂O₃ (obtained by treating γ-Al₂O₃ with HNO₃ and heating 4 hrs. at 450°) with a vol. rate of 1.23 yielded 85% dihydroxyran (XI), b₇ 60 86°, d₂₀ 0.923, sol. in H₂O (3% at room temp.) and in most org. compds. XI reacted easily with various substances like alcs., glycols, mercaptans, org. acids, and added Cl, H, HCl, COCl₂, or H₂O in the presence of traces of mineral acid XI with VIII yielded 85% product, b₁₅ 124-6°, d₂₀ 1.046, n_D 1.4591, a selective solvent of a few inorg. compds., and yielded with IV a liquid, b₁₂ 187-8°, d₂₀ 1.073, n_D 1.4622. XI heated with H₂O at 50° in the presence of traces of mineral acid yielded 87% 8-hydroxyvaleric aldehyde (XII), b₂ 51-2°, d₂₀ 1.053, n_D 1.4510, sol. in H₂O. XII hydrogenated over Cu-Cr catalyst at 130° under a pressure of 150 atm. yielded 92% 1,5-pentanediol (XIII), odorless viscous liquid, b₃ 119-20°, d₂₀ 0.989, n_D 1.4470. XI under a pressure of 40-60 atm. and at 110-15° in the presence of Ni over Cr oxide yielded 95% of tetrahydroxyran (XIV), b₇ 60 87-8°, d₂₀ 0.981, n_D 1.4211, sol. in H₂O (approx. 95% at 20°). In the vapor phase, the hydrogenation of XI under atm. pressure and at 120-30° with a vol. rate of 0.2-0.25 over skeletal Ni yielded only 85% XIV. XIV with SOCl₂ at 105-10° in the presence of ZnCl₂ yielded 50-55% 1,5-dichloropentane (XV) accompanied by much resin formation. XIV boiled with AcCl, 5 hrs., yielded 93-5% chloropentanol (XVI) acetate, b₁₅ 100-3°, d₂₀ 1.053, n_D 1.4360, which on being reesterified with MeOH yielded 94% of XVI, b₁₂ 98-9°, d₂₀ 1.049, n_D 1.4510. XVI with SOCl₂ at 130° yielded 80% XV, b₁₄ 69-71°, d₂₀ 1.093, n_D 1.4530; this roundabout way permitted increasing the yield of XV to 72% calcd. on XIV. The action of cyanides and alkali metals on XVI at 125° in aq. glycerol, 2 hrs., yielded 85% of the nitrile of hydroxycaproic acid, b₂₀ 150-2°, d₂₀ 0.970, n_D 1.4470, which was reduced in a NH₄OH-alc. soln. at 50° and 50-70 atm. with Raney Ni as catalyst to yield 73% aminohexanol, m. 50-1°, b₅ 118-20°. XIV oxidized by HNO₃ (d. 1.32) at a temp. below 25° yielded 87% glutaric acid, m. 97.5°, sol. in H₂O and alc. The action of Ac₂O on VII in the presence of AcOK at 135-40° yielded the K salt of furlacrylic acid (XVII); the K salt in its turn yielded 65% XVII, m. 139.5°, acid no. 401. Acetaldehyde was condensed with VII in 1% NaOH at 30° to yield 80% of the anhydride of XVII, m. 49-50°, b₁₀ 95-102°, which could not be oxidized to give the acid. A dry current of HCl was passed into an alc. soln. of XVII at 100° to yield the ester of oxopimelic acid (XVIII). Other esters (di-Et, di-Pr, di-Bu) were also obtained. The esters of XVIII sapon. more easily in an alk. medium than in an acid medium. The synthesis of II consisted in the removal of a carbonyl group from the mol. of VII at 400-20° over a mixt. of the oxides of Zn, Cr, and Mn in molar ratio 7:5:1 (mixed with graphite in the form of 4 + 4 mm. tablets) with a vol. rate of 0.3; simultaneously with VII water/vapor was added in the ratio 1:2.5; the reaction mixt. contained CO₂, H, and 95% II; the catalyst lost its activity after 50-5 hrs. and had to be regenerated; this was done in the same app. by blowing air 5-6 hrs. at a temp. not above 550°.

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 0.930, 1.4420, 333, 186, 2.4 + 1010, 0.2, -25°, XXIV, alc. with Cl₂, 220-5° (2), 0.915, 1.4499, 256, 225, 3.2 + 1011, 0.25, -30°, glutaric acid, XXVI, -, 0.926, 1.4465, 320.7, 181, 4.7 + 10, -, -35°, adipic acid, XXVI, -, 0.924, 1.4467, 301.7, 197, 8.7 + 1010, 0.5, -45°, adipic acid, VIII, -, 1.121, 1.4710(25), 364, 199, 2.3 + 109, 0.41, -35°, XVIII, XXVI, -, 0.961, 1.4530, 385, 197, 7.3 + 1010, 0.35, -50°, sebacic acid, VIII, -, 1.067, 1.4680(25), 298, 218, 7.3 + 109, 0.15, -25°, phthalic acid, VIII, -, 1.205(25), 1.5230, 320, 210, 4.4 + 109, 0.23, -, XX, XXVI, 237-9(3), 0.935, 1.4499, 256, 225, 3.2 + 1011, 0.25, -30°, C₇-C₉ acids, XX, 200-35° (5), 0.925, 1.4449, 312, 197, 4.5 + 1011, 0.07, -58°, C₇-C₉ acids, XXII, 220-90° (5), 0.936, 1.4482, 283, 212, 4.5 + 1010, 0.016, -50°, oleic acid, VIII, 222-7° (2), 0.922(25), 1.4655(25), 147-55, 196, 2 + 1011, 0.35, -50°, tetrahydrofurancarboxylic acid (XXVIII), XXVI, 117-20° (4), 0.9645, 1.4470, 244.2, -, -, -, XXVII, diethylene glycol, 216-18° (3), 1.1921, 1.4684, 376.6, -, -,
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 and a subsequent treatment with H. II was hydrogenated by bringing the reaction mixt. (without any previous sepn.) over molten Ni catalyst at about 120° with a vol. rate of 0.12, and cooling in dry ice to yield 90% III. After the sepn. of IV by simply cooling with H₂O, the gases were recirculated. VI formed an azetropic mixt. with H₂O (b. 63°) and contained 95% III. The ring of III was opened rather easily by the action of AcCl at 50°, upon cooling, to give 90% chlorobutanol acetate, b₃ 5 72-5°, d₂₀ 1.0852, n_D 1.4360; this, treated with AcOK at 160-70° yielded butanediol diacetate (XIX), b. 230°, d₂₀ 1.0460, n_D 1.4220. XIX could also be obtained in a 62% yield directly from III by the action of Ac₂O in the presence of H₂SO₄ at 93° (the temp. gradually rising to 145°) and the subsequent diast. of the excess Ac₂O and AcOH formed. XIX reesterified with MeOH in the presence of a small amt. HCl (3% on alc.) at 65-70° yielded AcOME and 90% butanediol (XX), m. 18.5°, b₇ 60 230°, d₂₀ 1.021, n_D 1.4460. The opening of the ring of III in the continuous process by the action of SOCl₂ and CoCl₂ at 100-2° yielded 30-80% V, b₁₃ 48-50°, d₂₀ 1.128, n_D 1.4520, and 60-14% dichlorobutyl ester, b₁₃ 126-8°, d₂₀ 1.0747, n_D 1.4568. V with alkali metal cyanides was converted at 140° in an aq. soln. (85%) of IV in the presence of a small amt. of XI into 81% adiponitrile, d₂₀ 0.9531, n_D 1.4340, which by sapon. in an alk. or an acid medium yielded 85% adipic acid, m. 150-1°. Hexamethylene diamine was obtained in a 95% yield by the hydrogenation of adiponitrile at 85-90° under a pressure of 100 atm. with a vol. rate of 0.3 over molten Co catalyst in a NH₃ alc. soln. The prepn. of ethers was accomplished by the interaction of V or VI with the dry Na salts of the synthetic fatty acids contg. 7-9 C atoms in a medium consisting of the same free acids at 180-90°, 14 hrs., by washing with acidified H₂O, and distg. The action of alkali metal cyanide on VI at elevated temp. in an aq. IV medium yielded 80% hydroxydivaleric acid (XXI) dinitrile, b₅ 175-80°, d₂₀ 0.963, n_D 1.4459. The alk. sapon. of XXI dinitrile yielded 77% XXI, m. 85-6°, and from XXI itself an ether b₃ 237-39°, d₂₀ 0.9353, n_D 1.4499, and sapon. no. 256, was obtained. The reduction of XXI dinitrile in an NH₃-alc. soln. at 100° over Raney Ni yielded 76% 5,5-di-(aminoamyl) ether, b. 135-7°, d₂₀ 0.9330, n_D 1.4627. VI heated with K phthalimide with the subsequent decompn. of the obtained product yielded 70% 4,4'-di(aminoamyl)ether, b₉ 125-6°, n_D 1.4568. VI treated with AcOK at 170-80° yielded 98% dibutylglycol (XXII) diacetate, b₄ 147-50°, d₂₀ 1.0253, n_D 1.4340, which reesterified with MeOH as above for XX yielded 92% XXII, b₄ 140-1°, d₂₀ 1.0041, n_D 1.4537. The substitution of one Cl in V by a cyano group in a soln. of adiponitrile at 135-40° yielded 62% chlorovaleronitrile, b₂₈ 115-17°, d₂₀ 1.0536, n_D 1.4430, which treated with Na₂S in an aq. soln. of IV at 115-20° yielded 70% thiodivaleric acid dinitrile (XXIII), b₃ 189-90°, d₂₀ 1.023, n_D 1.4868. The sapon. of XXIII in an acid medium yielded 75% thiovaleric acid, m. 94-5°. The oxidation of III by HNO₃ at below 25-30° yielded 90% succinic acid (XXIV), m. 183°. The oxidation under less severe conditions, e.g. in HNO₃ (d. 1.34) at 20-8° in C₆H₆ yielded 37% butyrolactone (XXV), b. 198-20°, d₂₀ 1.298, n_D 1.4350, and XXIV. XXV was also obtained by the dehydrogenation of XX over Cu-Cr catalyst at 230-40° (yield: 95%). The characteristics of a no. of complex esters obtained from the products of VII are given in the order: name of acid, name of alc., b.p., d₂₀, n_D, sapon. no., flash p., specific vol. resistance (ohm/cm.), losses on heating 6 hrs. at 100 (°), stability to freezing of the poly(vinyl chloride) films in degrees: XXIV, 2-ethylhexyl alc. (XXVII), 176-8° (25),

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 cf. C.A. 52, 6373b. Degradation of tylophorine (I) to 2,3,6,7-tetramethoxy-9-methylphenanthrene (II) in conjunction with other degradation and biogenetic considerations led to formulation of I as 2,3,6,7-tetramethoxyphenanthro[9,10-f]pyrrocoline. The basic phenanthro[9,10-f]pyrrocoline (III) was synthesized. I.MeI (2.5 g.) was converted to the methoxide and decomposed according to G., et al. (C.A. 49, 12500g), the product extracted with C₆H₆, the filtered extract evaporated to give 1.45 g. tylophorinemethine (IV), m. 185-8°, [α]_D 30D 0°, and the C₆H₆-insol. residue (0.4 g.) converted to tylophorine isomethoxide (V), m. 268-70° (decomposition), identified as tylophorine isomethoxyhydroxide (VI). IV (2 g.) by Hofmann degradation gave 0.15 to 0.75 g. de-N-methyltylophorinemethine (VII), m. 177°, and 0.65 g. non-basic alc., C₂₄H₂₆O₅ (VIII), m. 158-65°, benzoate, and 188-90° (MeOH). VII.MeI (0.84 g.) converted into the MeOH compound and decomposed in vacuo, extracted with C₆H₆ and the extract washed with dilute acid, the extract evaporated and the residue (0.26 g.) chromatographed in C₆H₆ over Al₂O₃, the column eluted with C₆H₆, and the product crystallized (MeOH) gave a colorless material, C₂₄H₂₄O₄ (IX), m. 181-8° (and not 152-3° as previously reported). Further elution with C₆H₆ containing 0.5% alc. gave 0.11 g. substance, sublimed at 170-240°/10-5 mm. to 12 mg. material, m. 88-115°, and 50 mg. amorphous residue, m. 230° (decomposition). VIII in all probability was formed by replacement of a NH₂ group by OH. The ultraviolet absorption spectra of I, IV, VII, and IX are closely similar to that of phenanthrene. I has no easily reducible unsatn. but IV can be converted to a quaternary iodide identical with V and it was concluded that IV has an 8-, 9-, or 10-membered ring system incorporating the N atom. IV (0.65 g.) in 100 ml. 99:1 alc. -AcOH hydrogenated 6 hrs. with 0.1 g. pre-reduced PtO₂, the filtered solution evaporated and the residue taken up in water, washed with C₆H₆, the aqueous acid solution made alkaline with Na₂CO₃, saturated with KCl and extracted with CHCl₃, the extract evaporated, and the residue recrystd. (alc.) gave I isomethochloride, m. 208-10°, identified by conversion to the known V. I (1 g.) in 50 ml. CHCl₃ added with stirring in 2 hrs. to 1.5 g. NBr in 25 ml. CHCl₃, kept overnight and evaporated, the residue triturated with dilute acid, filtered and the washed (NH₄OH and H₂O) residue recrystd. (C₆H₆) gave 1.05 g. bromocyanamide, C₂₄H₂₇N₂O₄.NBr (X), m. 163° (C₆H₆), with cleavage of one N-C bond in the alkaloid. Reduction of X under a variety of conditions failed to yield any pure product. X (1.05 g.) and 2.5 g. NaBH₄ in 150 ml. MeOH kept 24 hrs. with occasional shaking, the solution evaporated at 30° in vacuo, the washed (acid and water) and dried residue chromatographed in C₆H₆ on Al₂O₃, and the product crystallized (MeOH) gave 0.25 g. hydroxycyanamide, m. 195-8°, refluxed 3 hrs. in 5 ml. 4N H₂SO₄, the cooled solution washed with C₆H₆, and basified to give authentic I, indicating the presence of a 1,4- or 1,5-amino-alc. system in I. X (0.5 g.) in 50 ml. warm AcOH treated gradually with 2 g. Zn dust, the mixture boiled and filtered, the filtrate evaporated, the residue taken up in water, and the solution basified gave I, m. 283-5° (decomposition) (CHCl₃-alc.). X (1 g.) refluxed 3 hrs. with 15 ml. NH₄Et₂, the excess amine evaporated and the residue taken up in Et₂O, extracted with dilute acid and the acid layer basified, extracted with Et₂O, and the dried (Na₂SO₄) extract evaporated yielded 0.45 g. diethylamino compound, m.

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 157* (MeOH), refluxed 4 hrs. in 10 ml. 20% H2SO4, the soln. made alk. and extd. with Et2O, the ext. evapd., and the residue twice recrystd. (dil. MeOH) to give 0.27 g. urea deriv., C29H33N3O5, m. 199-200* (decompn.). The ready cleavage by NCB indicates the presence of a PhC2H2NH2 system in I and the high reactivity of X shows the presence of a CH2Br group in X. 1-Mel (3.5 g.) refluxed hrs. in 300 ml. 1:1 MeOH-H2O contg. 50 mg. KOH, the soln. concd. to 100 ml. and the cooled soln. filtered, the cryst. ppt. washed with ice water, and crystd. (H2O) gave 3.2 g. V. m. 268-70* (decompn.), λ 260, 288, 340, 355 m μ (log ϵ 4.81, 4.52, 3.39, 3.19). V (3.2 g.) refluxed 4 hrs. in 120 ml. 1:1 alc.-H2O contg. AgCl (from 10 g. AgNO3), the filtered soln. evapd. and the aq. soln. dild. to 100 ml., heated on a steam bath several hrs. with 100 g. 5% Na-Hg with occasional addn. of 100 g. Na-Hg, the decanted soln. filtered, and the dried residue recrystd. (C6H6-petr. ether) gave 1.4 g. isodihydroxyphenanthrene (XI), m. 200-2*, λ 300-40*, λ 260, 290, 340, 360 m μ (log ϵ 4.80, 4.54, 3.24, 3.12). XI (0.32 g.) in 45 ml. p-cymene dehydrogenated 4 hrs. with 0.5 g. 5% Pd-C at 220-40*, filtered, and the residue washed with C6H6, the combined filtrate and washings evapd., the residue triturated with dil. HCl, and the non-basic residue (0.25 g.) recrystd. (C6H6) gave tetrahydroisodihydroxyphenanthrene (XII), m. 235*, λ 255, 290, 340, 355 m μ (log ϵ 4.79, 4.54, 3.10, 2.90), giving pos. pine splinter and Ehrlich tests. XII (0.1 g.) in 30 ml. AcOH hydrogenated 7 hrs. with 0.1 g. prerduced PtO2 at 30*/60 lb./sq. in. with addn. of 0.05 g. catalyst after 4 hrs., the filtered soln. evapd. in vacuo, the residue taken up in 10 ml. 2N H2SO4 and washed with Et2O, the cooled acid layer basified, and the ppt. washed gave 0.08 g. XI. XI (0.95 g.) refluxed 4 hrs. with 4 ml. MeI in 50 ml. CHCl3, the ppt. washed (CHCl3), and crystd. (MeOH-Et2O) gave 1.25 g. XI. MeI salt, m. 278-9* (decompn.), λ 260, 290, 340, 355 m μ (log ϵ 4.85, 4.57, 3.25, 3.02). The salt (1.2 g.) in 120 ml. water shaken 4 hrs. with Ag2O (from 5 g. AgNO3), the filtered soln. evapd. at 50* in vacuo and the residue heated 1 hr. at 100*/0.5 mm. the product boiled in C6H6, and the filtered soln. evapd. gave 0.5 g. residue, m. 127-32*, chromatographed in C6H6 over 80 g. Al2O3, the column washed with 400 ml. C6H6 and 200 ml. C6H6 contg. 0.25% alc., eluted with 250 ml. C6H6 contg. 0.75% alc., and the fraction, m. 136-40*, recrystd. (Et2O) gave 0.25 g. isodihydroxyphenanthrene (XIII), m. 142*. XIII (0.2 g.) in 15 ml. Me2CO treated gradually with 1 g. KMnO4 in 200 ml. Me2CO, the soln. filtered after 72 hrs. and the residue digested with aq. Na2CO3, the filtered digest washed with CHCl3 and concd. the concentrate acidified and filtered, the pptd. acid (0.1 g.) taken up in MeOH and treated with CH2N2 (from 10 g. ONNMeCONH2), the mixt. kept 24 hrs. and evapd., the residue washed (dil. NH4OH-water), the solid (65 mg., m. 180-215*) extd. with 10 ml. boiling MeOH, and the filtered ext. cooled gave 8 mg. Me-9-methyl-2,3,6,7-tetramethoxy-10-carboxylate (XIV), m. 185-6*, λ 260, 290, 340, 358 m μ (log ϵ 4.75, 4.54, 3.40, 3.15). Recrystn. of the MeOH-insol. fraction from C6H6 yielded 8 mg. di-Me-9-methyl-2,3,6,7-tetramethoxyphenanthrene-9,10-dicarboxylate (XV), m. 246-7*, λ 260, 290, 330 m μ (log ϵ 4.61, 4.45, 3.26). Sepn. of the 2 esters (150 mg.) by chromatography over Al2O3 and elution with 1:999 MeOH-C6H6 and 1:199 MeOH-C6H6 gave 90 mg. XIV and 5 mg. XV. Oxidation of XIII gave generally only XV. XIV (50 mg.) refluxed 8 hrs. in 10 ml. alc. with 2 g. KOH, the solvent evapd. and the residue taken up in hot water, the filtered soln. acidified and the acid (40 mg., m. 225-7*) heated 3 hrs. in 2 ml. quinoline with 20 mg. CuSO4.5H2O, the cooled mixt. treated with 100 ml. C6H6 and washed successively with dil. acid, water, dil. NaOH, water, the dried (Na2SO4)

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 in vacuo gave 20 mg. II, m. 188-9*, λ 255, 285, 340, 355 m μ (log ϵ 4.74, 4.41, 3.19, 2.99). 2-Amino- α -(6-bromo-3,4-dimethoxyphenyl)-3,4-dimethoxyphenanthrene acid (II g.), prepd. according to Kondo and Ochiai, C.A. 23, 3709) diazotized with 6 ml. iso-AmNO2 and treated with NaH2PO2.H2O-Cu mixt. gave 5 g. product, fractionally crystd. (MeOH) to give 1 g. more sol. 8-bromo-3,4,5,6-tetramethoxyphenanthrene-9-carboxylic acid, m. 187*, and 2 g. less. sol. 3,4,5,6-tetramethoxyphenanthrene-9-carboxylic acid (XVII), m. 236*. XVII (2.5 g.) refluxed 2 hrs. in 25 ml. anhyd. MeOH contg. 1 ml. concd. H2SO4, the MeOH evapd. and the residue dild. with water, the soln. extd. with CHCl3, and the washed and dried ext. evapd. gave 1.5 g. XVII Me ester, m. 70*, refluxed 4 hrs. with 15 ml. 64% N2H4.H2O in 15 ml. abs. alc. to yield 1.3 g. XVII hydrazide, m. 175* (C6H6). The hydrazide (1.1 g.) was converted with 0.6 g. PhSO2Cl in 15 ml. C5H5N to N-benzenesulfonyl-3,4,5,6-tetramethoxyphenanthrene-9-carboxylic acid hydrazide, m. 217*, rearranged in 25 ml. (CH2OH)2 with 0.6 g. Na2CO3 to 0.3 g. 3,4,5,6-tetramethoxyphenanthrene-9-aldehyde, m. 121* (alc.). The aldehyde (0.3 g.) 0.6 ml. anhyd. N2H4, and 6 ml. alc. refluxed 2 hrs. the solvent evapd. in vacuo, the residue heated 25 min. at 120-5* with 0.3 g. powd. KOH, the product suspended in water and extd. with Et2O, the ext. evapd. and the residue filtered in C6H6 through Al2O3, the eluate evapd., and the residue crystd. (MeOH) gave 80 mg. 3,4,5,6-tetramethoxy-9-methylphenanthrene, m. 87-8*, λ 265, 310, 325, 368, 380 m μ (log ϵ 4.73, 4.16, 4.16, 3.43). Subjecting 15 g. 2-amino- α -(3,4-dimethoxyphenyl)-3,4-dimethoxyphenanthrene to the Paschor reaction gave 2.5 g. 3,4,6,7-tetramethoxyphenanthrene-9-carboxylic acid, m. 210*; Et ester m. 105*. The ester (0.6 g.) was converted through 0.5 g. hydrazide, m. 219* (alc.), to 0.25 g. benzenesulfonyl deriv., m. 245* (alc.-dioxane), and rearranged to 0.125 g. 3,4,6,7-tetramethoxyphenanthrene-9-aldehyde, m. 148* (alc.), reduced by the Wolff-Kishner method to 3,4,6,7-tetramethoxy-9-methylphenanthrene, m. 123* (MeOH), λ 260, 315, 345, 360 m μ (log ϵ 4.70, 3.86, 3.21, 3.10). The identification of II provided pos. evidence as to the presence of a phenanthrene ring, the orientation of the MeO groups, and one point of attachment of the heterocyclic system in I. I (1.3 g.) in 20 ml. water stirred with 210 ml. 5% KMnO4 with warming as the rate of oxidation slackened, the residue on filtration extd. with CHCl3 and the concd. ext. filtered through Al2O3, the eluate (100 ml.) evapd., and the residue recrystd. (CHCl3-alc.) gave 10 mg. needles, C20H20O7, m. 249*. Further elution with CHCl3 gave 10 mg. brown needles, m. 245-5*. No acidic material was recovered from the original filtrate. VI (1 g.) in 25 ml. alc. and 15 ml. C5H5N treated with 0.1N KMnO4 at 30* and with warming to persistence of pink color, the filtered soln. evapd. in vacuo and acidified, the gummy product treated with aq. NaHCO3 and washed with Et2O, the aq. soln. acidified, and the ppt. crystd. (AcOH) gave 6 mg. XVIIa. The oxidation residue extd. with hot CHCl3, the washed (dil. acid) and dried ext. evapd., and the residue crystd. (AcOH or C5H5N) gave 12 mg. 2,3,6,7-tetramethoxyphenanthrene-9,10-dicarboximide, m. 355* (decompn.). V (0.5 g.) in 150 ml. water stirred at 30* and finally at 100* with 130 ml. 3% KOH, the filtered and the residue washed thoroughly with hot water, the filtrates evapd. and the residue taken up in dil. H2SO4, the soln. extd. 12 hrs. with Et2O and the dried (Na2SO4) ext. evapd., the residue from 2 runs sublimed at 180-200* in a high vacuum, the sublimate (0.1 g.) taken up in 5 ml. alc. and 2 ml. EtNH2, the soln. evapd. and the residue taken up in alc. EtNH2, the solvent evapd. and the residue heated 5 min. at 180*, the product sublimed at

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 C6H6 layer evapd., the residue filtered in C6H6 through Al2O3, the eluate evapd., and the residue recrystd. (MeOH) gave 10 mg. II, m. 188-9*, λ 255, 285, 340, 355 m μ (log ϵ 4.88, 4.50, 3.20, 2.97). XV (0.12 g.) refluxed 4 hrs. with 2 g. KOH in 10 ml. alc., the soln. dild. with 50 ml. water and the alc. evapd., the cooled aq. soln. washed with Et2O, and acidified gave 100 mg. 2,3,6,7-tetramethoxyphenanthrene-9,10-dicarboxylic acid anhydride (XVI), m. 315-25* (decompn.). The identification of II was rendered easier by considering certain biogenetic aspects and the choice of possible tetramethoxyphenanthrenes was further aided by synthesis according to Buchanan, et al. (C.A. 38, 58202), from the corresponding 9-carboxylic acids. Ac2O (40 ml.), 21 g. 6,3,4-O2N(MeO)2C6H2CHO, 21 g. 3,4-(MeO)2C6H3CH2CO2H, and 20 ml. NEt3 heated (dry atm.) 16 hrs. at 100*, the cooled mixt. dild. with 20 ml. water, kept for some time and poured into 1 l. water contg. 160 g. K2CO3, the mixt. heated and the cooled red soln. washed twice with 100 ml. Et2O, the soln. acidified with HCl, and the gummy product crystd. (MeOH) gave 28 g. 3,4-dimethoxy- α -(3,4-dimethoxyphenyl)-6-nitrocinnamic acid, m. 185*. The nitro acid (10 g.) in 180 ml. 4N NH4OH added with stirring to 180 ml. concd. NH4OH (d. 0.88) and 75 g. FeSO4.7H2O in 180 ml. hot water, the mixt. stirred 45 min. at 90* and the cooled filtrate washed with C6H6, carefully neutralized with concd. HCl, the pptd. amino acid washed (cold MeOH and water) and dried, the acid (3 g.) in 90 ml. dioxane and 1 ml. concd. H2SO4 at 30* heated dropwise with stirring with 2 ml. freshly prepd. iso-AmNO2 in 10 ml. dioxane, the diazonium soln. added with stirring at 40* to 1 g. freshly ppt. Cu and 20 g. NaH2PO2.H2O in 20 ml. water, the stirring continued several hrs. at 40* and 1 hr. at 90*, the mixt. poured into 1 l. water contg. sufficient NH4OH to dissolve the acidic product, the filtered soln. acidified with concd. HCl and the ppt. boiled 3 times with 20 ml. MeOH, the insol. residue taken up in dil. Na2CO3, the washed (C6H6) soln. filtered, and acidified gave 0.4 g. 2,3,6,7-tetramethoxyphenanthrene-9-carboxylic acid (XVIIa), m. 285*. The MeOH filtrate evapd. and the residue crystd. (Me2CO) yielded 0.3 g. 3,4,6,7-tetramethoxyphenanthrene-10-carboxylic acid, m. 210*, decarboxylated to authentic 3,4,6,7-tetramethoxyphenanthrene, m. 121*. XVIIa (0.1 g.) refluxed 1 hr. in 3 ml. quinoline with 0.1 g. CuSO4, the cooled mixt. poured into excess HCl and extd. 3 times with 25 ml. C6H6, the washed (dil. alkali and water) and dried (Na2SO4) ext. evapd., and the residue crystd. (MeOH) gave 30 mg. 2,3,6,7-tetramethoxyphenanthrene, m. 178*, λ 255, 285, 340, 350 m μ (log ϵ 4.86, 4.38, 4.16, 3.08, 2.78). XVIIa (2 g.) in 20 ml. MeOH treated 24 hrs. with CH2N2 (from 20 g. ONNMeCONH2) in Et2O yielded 1.8 g. XVI Me ester, m. 195* (alc.). The ester (0.5 g.), 1.5 ml. 85% N2H4.H2O, and 5 ml. iso-AmOH refluxed 4 hrs., the cooled mixt. treated with 20 ml. MeOH, and the product crystd. (alc.) gave 0.3 g. XVIIa hydrazide, m. 246* (decompn.), dried at 140* in vacuo, kept 24 hrs. at 0* in 10 ml. C5H5N with 0.4 g. PhSO2Cl, poured into ice and HCl, and the ppt. recrystd. (Me2CO-MeOH) to give 0.2 g. benzenesulfonyl deriv., m. 265* (decompn.). The deriv. (0.2 g.) dried at 140* in vacuo treated 80 sec. in 10 ml. HOCH2CH2OH at 160* with 0.2 g. anhyd. Na2CO3, dild. with warm water and the cooled mixt. extd. with CHCl3, the water-washed and dried (Na2SO4) CHCl3 layer evapd., and the residue crystd. (AcOH) gave 0.15 g. 2,3,6,7-tetramethoxyphenanthrene-9-aldehyde, m. 210*. The aldehyde (0.1 g.) refluxed 2 hrs. with 1 ml. 85% N2H4.H2O in 5 ml. abs. alc., the solvent evapd. in vacuo and the residue heated 30 min. at 190* with 0.2 g. powd. KOH, the mixt. dild. with 10 ml. water and the soln. extd. with C6H6, the ext. washed (H2O) and dried (Na2SO4), the ext. evapd., and the residue sublimed

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 150-200* in vacuo and the sublimate crystd. (alc.), the cryst. product (30 mg., m. 225-7*) filtered in alc. through Al2O3, and the fraction recrystd. (alc.) gave authentic N-ethyl-m-hemipinimide, m. 230-1*. IV (0.5 g.) in 25 ml. dry C5H5N treated gradually with 1 g. KMnO4, the mixt. filtered and the filtrate evapd., the residue washed successively with acid, alkali, and water, and the neutral product (0.35 g.) crystd. (C6H6-petr. ether) gave a compd., C25H27NO5, m. 241-4*, apparently formed by oxidation of a CH2 group to a CO group, confirmed (by reduction with LiAlH4 to IV. IV (0.7 g.) in 25 ml. tetrahydrofuran basified with concd. aq. NaOH and stirred at 100* with 365 ml. 30% H2O2, the soln. concd. to 30 ml. in vacuo and washed with Et2O, the aq. layer acidified and extd. 10 times with 150 ml. Et2O, the ext. evapd., and the acid (10 mg.) esterified with CH2N2 in Et2O gave XV, m. 247-8* (C6H6-MeOH). On the basis of these degradations the constitution of I was assigned and III was synthesized as a model for the synthesis of I. Powd. AgNO3 (48 g.) added with stirring in 5 hrs. to 78 g. Br(CH2)4OC2Ph (Cloke and Pilgrim, C.A. 34, 4233), the mixt. heated 4 hrs. at 100*, the cooled product taken up in Et2O, the filtered ext. evapd., and the residue fractionated gave 8-nitrobutyl benzoate, b.p. 156-7*. Phenanthrene-9-carboxaldehyde (10 g.), 10 ml. benzoate, and 5 g. NH4OAc refluxed 2 hrs. in 60 ml. AcOH, the red soln. poured into water and the water-washed oil taken up in alc., filtered, and the solid recrystd. (alc.) gave 5-benzoyloxy-2-nitro-1-(9-phenanthryl)pentane (XVIIa), m. 118*. XVIIa (5 g.), 200 ml. AcOH, 50 ml. alc., and 15 ml. concd. HCl stirred in the cathode compartment of a cell with a Pb electrode, a steady current of 15 amp. passed 18 hrs. with 20% H2SO4 in the anode compartment, the catholyte filtered and evapd. in vacuo, the residue triturated with aq. NH4OH and extd. with Et2O, the product refluxed 2 hrs. in 70 ml. 4N H2SO4, the cooled soln. washed with Et2O, basified with NH4OH, and extd. with Et2O gave 4-amino-5-(9-phenanthryl)pentanol (XVIII), m. 120* picrate m. 175* (alc.). XVIIIa (8.5 g.) in 50 ml. dry tetrahydrofuran added (dry atm.) with stirring to 3 g. LiAlH4 in 30 ml. tetrahydrofuran, the mixt. decompd. with water and the filtered soln. concd., the concentrate dild. with Et2O, and cooled to 10* gave 1.2 g. 3-(9-phenanthrylmethyl)-1,2-oxazine, m. 185*, λ 255, 300, 350 m μ (log ϵ 4.73, 4.03, 2.56). The mother liquor kept at 0* overnight and filtered gave XVIII; mono-Bz deriv. m. 155*. XVIII (1 g.) heated 2 hrs. at 180* with 3 ml. 98% HCO2H and the neutral product isolated gave 1 g. O,N-diformyl deriv., m. 145*, refluxed 1 hr. with 10 ml. 10% aq. NaOH, the water-washed gummy product triturated with C6H6, and the solid recrystd. (large vol. C6H6) to give 0.6 g. 4-formamido-5-(9-phenanthryl)pentanol (XIX), m. 150*. XIX (0.5 g.) refluxed 1 hr. in 5 ml. 5-free dry PhMe with 5 ml. POCl3, the PhMe layer dild. with excess petr. ether, the gummy ppt. washed with petr. ether and extd. with hot water, the cooled neutralized aq. ext. treated with excess KI, the quaternary iodide (0.2 g.) washed and hydrogenated in MeOH with 50 mg. prerduced PtO2 at 50 lb./sq. in., the filtered soln. evapd. and the residue ground with alkali, the mixt. extd. with CHCl3, and the product recrystd. (MeOH) gave 50 mg. III, m. 170*. A 255, 270, 300, 335, 350 m μ (log ϵ 4.73, 3.56, 3.48, 2.6, 2.6, 2.68). Formulations for IV, XI and XII are postulated.
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AB (Y throughout this abstract = the 4-methylxanthone nucleus, Z = the 4-methylthiexanthone nucleus). The title compds. are prepared by the following methods. (1) Reaction of bases containing at least one primary and one secondary N atom with substituted 4-methylxanthones (I) or 4-methylthiexanthones (II), containing in the 1-position a reactive radical, above 100° in the presence or absence of solvents and preferably in the presence of catalysts (Cu bronze), and reduction of the reaction products to xanthidrols or thiexanthidrols, e.g. with Zn powder and alkaline hydroxides. (2) Reaction of xanthones, xanthidrols, thiexanthones, thiexanthidrols, containing an Me group in 4-position and an NH₂ group or formylamino group in the 1-position with basically substituted alcs. or esters thereof, e.g. with halides, alkyl and aryl sulfonates or salts thereof, in the presence of a condensation catalyst and then splitting off the formyl radical. (3) Condensation of 2-methyldiphenyl ethers or thioethers substituted in 5-position by a basically substituted NH₂ group and in 2-position by a CO₂H group or derivs. thereof. (4) Condensation of 2-amino-5-methylbenzophenones, the NH₂ group being substituted by a basic radical, substituted in each ring by a OH or SH group or in one ring by a OH or SH group and in the other ring by a halo atom in o-position to the carbonyl group, in the presence of condensation agents (acids and alkali hydroxides). Condensation of o-ClC₆H₄CO₂H (III) with 5,2-ClMeC₆H₃OH (IV) yields 2-carboxy-5'-chloro-2'-methyldiphenyl ether, m. 117-18° (alc.). Cyclization with concentrated H₂SO₄ yields 1-ClY, m. 133-4° (alc.). Heating 1-ClY 24.5 with ethylenediamine 12 and pyridine 18 in the presence of some Cu bronze 6 hrs. to 160-70°, cooling, adding N NaOH 100 to the mixture, steam distilling, dissolving the yellow distillation residue in dilute AcOH, and precipitating the free base by a solution of NH₃ in CH₂Cl₂ yields 1-HZNCH₂NH₂ 17 parts, yellow, m. 140-1° (alc.). Similarly are prepared 1-EtZnCH₂CH(OH)CH₂NH₂ (hydrochloride, yellow, m. 181-2°) and 1-EtZnCH₂CH₂CH₂OMeY as the yellow salt of methylenebis(2-hydroxy-3-naphthoic acid), insol. in H₂O, decompose 224°. 1-PhOY 30.2, prepared by reaction of 1-ClY and PhONa in PhOH, m. 130-1°, and EtZnCH₂CH₂NH₂ (V) 36 is heated 6 hrs. in a tube to 170°, the excess amine is removed by steam-distillation after addition of N NaOH 100 parts, and the yellow residue dissolved in 10% AcOH and treated with dilute NaOH to give 1-EtZnCH₂CH₂NH₂ (VI), yellow, m. 76-7° (alc.); hydrochloride, yellow, m. 190-1° (alc.). The same compound is prepared by heating 1-BrY 28.9, m. 130-1° (alc.), prepared from 2-carboxy-5'-bromo-2'-methyldiphenyl ether by ring-closure with concentrated H₂SO₄, with H₂N(CH₂)₂OH 25 and pyridine 10 parts in the presence of some Cu bronze 6 hrs. in a tube to 170°, treating the formed 1-HOCH₂CH₂NH₂, yellow, m. 187-8° (alc.), with excess POC₃ 2 hrs. on the water bath, removing the excess POC₃ by distillation, decomposition of the red residue with NH₃ under ice-cooling, and purification of the 1-ClCH₂CH₂NH₂ by recrystn. from alc., yellow needles, m. 145-6°, and reaction with NHEt₂. Similarly are prepared (substituents in 1 position, derivs., and m.ps. given): EtNHCH₂CH₂NH₂, hydrochloride, 226-7°; piperidylethylamino, yellow crystals from ligroine, 90-1°, hydrochloride, yellow crystals, 265° (decomposition), III and 5,2-bromo-6-hydroxy-3-methoxy-2'-methyldiphenyl ether. VI may also be prepared by condensation of o-ClC₆H₄CO₂Na with 2,5-Me(EtZnCH₂CH₂NH₂)C₆H₃ONa (the free phenol b4 178-9° and is

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prep. by demethylation of its Me ether, b5 162-4°, with HBr) and treatment of the brown crude 2-carboxy-5'-diethylaminoethylamino-2'-methyldiphenyl ether with concd. H₂SO₄ 1 hr. on the water bath. VI may also be prep. by heating 10% AcOH and 2,2'-dihydroxy-3-methyl-6-diethylaminoethylaminobenzophenone, m. 88-9° (picrate, m. 177-8°), prep. by refluxing VI with alc. KOH in a tube. Hydroxymethylxanthone is formed as by-product, m. 148-9°. Reduction of VI 20 with Zn powder 50 in the presence of NaOH 40 and alc. 320 parts under reflux yields 1-diethylaminoethylamino-4-methylxanthidrol, m. 100° (ligroine, b. 60-70°). Reaction of 1,6-Cl₂Y 27.9, m. 177-8° (glacial AcOH), prep. by cyclization of 2-carboxy-5,5'-dichloro-2'-methyldiphenyl ether, m. 175°, with concd. H₂SO₄, with V 36 parts at 170° yields 6,1-Cl(EtZnCH₂CH₂NH₂)Y yellow, m. 87-8° (ligroine); hydrochloride, m. 255-6° (decomp.). This product may also be prep. by heating 6,1-Cl(O₂N)Y 29 parts, m. 256-8°, with V to 170-80°. 6,1-Cl(O₂N)Y is prep. by cyclization of 2-carboxy-2'-methyl-5'-nitro-5-chlorodiphenyl ether, m. 198°, prep. by condensation of 2,4-HO(O₂N)C₆H₃Me with 2,4-Cl₂C₆H₃CO₂H. Similarly are prep.: 6,1-Cl(EtZnCH₂CH(OH)CH₂)Y, oily base; 6,1-Cl(EtZnCH₂CH₂CH₂OMe)Y, semicryst. base. Heating 1,7-Cl₂Y 27.9, m. 198°, and V 36 in a tube to 170° in the presence of a little Cu bronze yields 7,1-Cl(EtZnCH₂CH₂NH₂)Y 32 parts, yellow, m. 243° (dark coloration). 1,7-Cl₂Y is prep. by cyclization of 2-carboxy-4,5'-dichloro-2'-methyldiphenyl ether, yellow, m. 177-8° (alc.), prep. by condensation of 2,5-Cl₂C₆H₃CO₂H with IV, with concd. H₂SO₄. Reaction of 2,4-Cl(MeO)C₆H₃CO₂H with IV yields 2-carboxy-5'-chloro-2'-methyl-5-methoxydiphenyl ether, m. 174-5° (alc.). Heating with AlCl₃ yields 1,6-Cl(MeO)Y, m. 176-7° (alc. or glacial AcOH). Heating this product with V 6 hrs. to 160-70° in a tube yields 6,1-MeO(EtZnCH₂CH₂NH₂)Y, yellow, m. 84-5° (ligroine); hydrochloride, yellow, m. 225-6°. Condensation of 2,5-Cl(MeO)C₆H₃CO₂H and IV yields 2-carboxy-5'-chloro-2'-methyl-4-methoxydiphenyl ether, m. 183°. Cyclization with AlCl₃ yields 1,7-Cl(MeO)Y, m. 175-6° (alc.). Heating with V 6 hrs. in a tube to 160-70° in the presence of a little Cu bronze affords 7,1-MeO(EtZnCH₂CH₂NH₂)Y; hydrochloride, yellow, m. 189-90° (alc.). Similarly is prep. 7,1-MeO(HZNCH₂CH₂NH₂)Y, yellow, m. 157-8° (alc.). Reaction of 2,6-Cl(MeO)C₆H₃CO₂H with IV yields 2-carboxy-5'-chloro-2'-methyl-3-methoxydiphenyl ether, m. 178-9° (alc.). Heating this product with V to 180° in the presence of Cu bronze yields 8,1-HO(EtZnCH₂CH₂NH₂)Y; hydrochloride, yellow, m. 226°. Reaction of 2,4-ClMeC₆H₃CO₂H with IV yields 2-carboxy-5'-chloro-2',5'-dimethyldiphenyl ether, m. 138-9° (alc.). Cyclization with concd. H₂SO₄ gives 1,6-ClMeY, m. 168° (alc.). Heating this substance with V to 170-80° yields 6,1-Me(EtZnCH₂CH₂NH₂)Y; hydrochloride, yellow crystals after purification via the acetate, m. 217-18°. 2,5-ClMeC₆H₃CO₂H is treated with IV to give 2-carboxy-5'-chloro-2',4'-dimethyldiphenyl ether, m. 173-4° (alc.) after sintering. Cyclization with concd. H₂SO₄ yields 1,7-ClMeY, m. 152° (alc.). Heating this product with V yields 7,1-Me(EtZnCH₂CH₂NH₂)Y, yellow, m. 82°; hydrochloride, yellow, m. 198°. Heating 24 g. 1-HZNZ and 15 g. V to 150° (alternatively 12 g. 1-HZNZ with 12 g. EtZn(CH₂)₂OH to 180-90° in the presence of 7.1 g. P₂O₅) yields 21 g. 1-EtZnCH₂CH₂NH₂, yellow, m. 64-5°; hydrochloride, m. 195-6°. Reaction of 4,4'-dichlorodithiosalicylic acid and p-ClC₆H₄Me yields a mixt. of 1,6-Cl₂Y and 4,6-dichloro-1-methylthiexanthone, m. 182-3°. Heating this mixt. with V to 170-80° yields 6,1-Cl(EtZnCH₂CH₂NH₂)Z, yellow,

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m. 96-7° (alc.); hydrochloride, yellow, m. 246-7° (MeOH). Treatment of thiosalicylic or dithiosalicylic acid with p-ClC₆H₄Me in the presence of concd. H₂SO₄ yields a mixt. of 1-Cl₂ and 1-methyl-4-chlorothiaxanthone, m. 145-6°. Heating with V yields 1-EtZnCH₂CH₂NH₂. Similarly are prep. 2 derivs. (substituents in 1-position, derivs., and m.ps. given): EtZnCH₂CH(OH)CH₂NH₂, 99-100°, hydrochloride, 176-7°; EtZnCH₂CH₂CH₂NH₂, hydrochloride, 173°; EtZn(CH₂)₂NH₂, hydrochloride, 175°. 1-EtZnCH₂CH₂NH₂ is also prep. by heating a soln. of NaOEt (prep. by dissolving 2.3 g. Na in 50 cc. alc.) with 15.8 g. EtZnCH₂CH₂NH₂CHO and 52 g. 1-Cl₂ in the presence of 75 g. decahydronaphthalene 4 hrs. to 180°. EtZnCH₂CH₂NH₂CHO, b3 105°, is prep. by heating V with HCO₂Et for several hrs. The reaction products are useful as anthelmintics. Cf. C.A. 52, 15592i.

ACCESSION NUMBER: 1959:2142 CAPLUS
DOCUMENT NUMBER: 53:2142
ORIGINAL REFERENCE NO.: 53:411c-i, 412a-i, 413a
TITLE: Xanthenes and thiexanthones
INVENTOR(S): Mauss, Hans
PATENT ASSIGNER(S): Farbenfabriken Bayer A.-G.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 919107		19541014	DE	

L20 ANSWER 202 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Accelerators consisting of an onium compound and a small proportion of a heavy metal compound are used in the polymerization of organic compds. containing terminal or intermediate C=C bonds and organic S compds., such as sulfinic acid or its salts, a sulfonylate, an α -amino sulfone, or a mercaptan. The onium compound is an organic ammonium, sulfonium, or oxonium compound; most effective are salts of quaternary ammonium bases. The O for polymerization is added either as a peroxide or as atmospheric O. The addition of small amts. of monohydric or polyhydric aliphatic alcoh. increases the activity of the accelerators. For example, 1.3 g. poly(methyl methacrylate) and 0.75 ml. mono(Me methacrylate) were polymerized at an initial temperature of 22-3° with 0.5% dodecylbenzylammonium chloride, 2% MeC₆H₄SO₂CH₂OH, 2% MeOH, 10% Cu⁺⁺, and atmospheric O, a polymerization time of 7 min. being required.

ACCESSION NUMBER: 1958:108842 CAPLUS
 DOCUMENT NUMBER: 52:108842
 ORIGINAL REFERENCE NO.: 52:192401, 19241a-b
 TITLE: Onium compounds as polymerization accelerators
 PATENT ASSIGNER(S): Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 792812		19580402	GB	

L20 ANSWER 203 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 (XI), m. 162-3°, identical with the sample obtained from the hydrogenation of IX. XI acetate did not depress the m.p. of the acetate from the hydrogenation of IX. VIII (15 g.) in 150 ml. alc. hydrogenated over 20 g. of the same catalyst at an initial pressure of 2500 lb./sq. in. and temp. raised to 100°, held until no more H was absorbed, and the product extd. in a Soxhlet extractor gave 10.5 g. X. IX (5.2 g.) in 100 ml. methylcyclohexane hydrogenated over 23 g. of catalyst as above with a final temp. of 250° at 4200 lb./sq. in. gave XI. XI acetate m. 142-3°. The methylcyclohexane filtrate from XI was concd. and treated with ligroine to give C13H21NOMe₂ (XII), m. 79-80°, HCl salt, m. 212-14°. Another hydrogenation of 12 g. IX at a final temp. of 270-80° and 4000 lb./sq. in. gave 3.4 g. XI, 2 g. of an unidentified acid sol. oil, and 2 g. compd., C14H26O, b₂ 122-3°, m. 56-7° (phenylurethan (XIII), m. 155-6°, mixed m.p. with the phenylurethan of authentic 2-methyl-6-(cyclohexanemethyl)cyclohexanol (m. 138-9°), 118-21°). XI (6.7 g.), 9 g. MeI, and 1.5 g. KOH left 2 days at room temp. with 200 ml. alc., the sepd. XI removed, and the filtrate concd. gave the quaternary salt (XIV), m. 186-7° (alc.-Et₂O). XIV obtained from XII was identical. IX (17 g.) in 150 ml. MeOH was hydrogenated over 60 g. catalyst and an initial pressure of 2600 lb./sq. in. There was a large drop in pressure at 110°, the final temp. was 230°. The alc. soln. dild. with Et₂O, extd. with dil. HCl, the acid ext. made alk., and the product sepd. gave 9.3 g. XII. Evapn. of the Et₂O layer gave 1.5 g. compd., C13H20NO.CH₂OH, m. 190-1° (alc.). IX (9 g.) and 0.4 g. 5% Pd-C was heated to 200° under H and the receiver placed in a Dry Ice bath. At 275° the evolution of gas was vigorous and some BaCO₃ sepd. in the trap; when gas was no longer evolved, the temp. was raised to 210° and held there 6 hrs. The residue extd. with alc. and decolorized with C gave X. Cyclopentanone (89 g.), 55 ml. MeNO₂, and 200 ml. C₆H₆ refluxed 24 hrs. gave 6.5 g. compd. (XV), C12H16N₂O₃, m. 242-5° (decompn.) (alc.), purple color test with FeCl₃, its solubilities the same as for I. XV (2.5 g.) refluxed 1 hr. with 100 ml. 1:4 H₂SO₄ gave 1.7 g. compd., C12H15NO₃, m. 157-8° (alc.-ligroine). 4-Methylcyclohexanone (112 g.), 61 g. MeNO₂, and 6 ml. IV refluxed 30 hrs. gave 10.5 g. compd. (XVI), m. 257-8° (decompn.) (EtOCH₂CH₂OH). Hydrolysis of 2 g. XVI by refluxing for 50 hrs. with 160 ml. 1:4 H₂SO₄ gave a compd., C16H22NO₃, m. 160-2°.

ACCESSION NUMBER: 1958:72158 CAPLUS
 DOCUMENT NUMBER: 52:72158
 ORIGINAL REFERENCE NO.: 52:12799c-i, 12780a-f
 TITLE: Reaction of nitroparaffins with alicyclic ketones. III. The solid by-product from nitromethane and cyclohexanone
 AUTHOR(S): Nightingale, Dorothy V.; Reich, Donald A.; Erickson, Floyd B.
 CORPORATE SOURCE: Univ. of Missouri, Columbia
 SOURCE: Journal of Organic Chemistry (1958), 23, 236-41
 CODEN: JOCEAH; ISSN: 0022-3263
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 203 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB cf. C.A. 47, 6877b. The solid by-product (I) from the reaction of MeNO₂ (II) with cyclohexanone (III) in the presence of piperidine (IV) or secondary amine catalysts may be a heterocyclic hydroxamic acid containing N and O in the heterocyclic system. Other functional groups believed to be present are -C=C- and -C=N- not conjugated. The chemical reactions and infrared absorption spectra of this solid and compds. derived from it are consistent for the most part with a structure containing these functional groups. III (180 ml.), 102 ml. II, 200 ml. C₆H₆, and 12 ml. IV refluxed gently 30 hrs., cooled, and the solid collected gave 33 g. I, m. 262-3° (decomposition) (EtOCH₂CH₂OH). C14H20N₂O₃. The H₂O collected in the trap had a pH of 8-9; acidification and testing with starch iodide paper indicated HNO₂ being developed. Evaporation of the H₂O gave a solid which gave a pos. brom ring test with FeSO₄ and H₂SO₄, and liberated NH₃ when treated with NaOH. I diacetate m. 128-9°. I (10 g.), 75 ml. alc., and 4.5 g. KOH treated dropwise with 6.7 g. BrCH₂CO₂Et under reflux, the solution refluxed 6 hrs., cooled, diluted, and the product collected gave 10 g. C13H18NO(CO₂CH₃CH₂CO₂Et) (V), m. 163-5° (aqueous alc.). Hydrolysis of V with dilute HCl gave C13H18NO(CO₂CH₂CO₂H), m. 137-8°. I (2 g.) in 50 ml. CCl₄ refluxed with excess Br gave the monobromo compound, m. 184-5°. I (5 g.) pyrolyzed at 280-50° gave a white solid and a brown liquid; they were extracted with Et₂O and the solid collected. This solid reacted with PhSO₂Cl to give a benzenesulfonamide, m. 150.5-2.5°. Treating an aqueous solution of the solid with HCl gave a gas which indicated that the solid was (NH₄)₂CO₃. The Et₂O extract dried and the solvent removed gave 7 g. residue which was distilled to give 2.2 g. cyclohexyl cyanide (VI), b₁₀ 65-6° n_D20 1.4575. Hydrolysis of VI in concentrated H₂SO₄ gave cyclohexanecarboxamide, m. 184.5-6.0°. I (13.2 g.) in 100 ml. alc. was hydrogenated over 3 g. Raney Ni at an initial pressure of 2900 lb./sq. in.; after heating began there was a large pressure drop at 105-30°. Heating continued to 160° and 4240 lb./sq. in., the catalyst removed, and the solvent distilled gave 10.9 g. C13H20NO(CH₂NH₂) (VII), m. 192-3° (ligroine). Hydrogenation of 16 g. I in 170 ml. alc. over 20 g. Cu chromium oxide at 90-110° and 3400 lb./sq. in. gave 12 g. VII; monoacetyl derivative (by refluxing 10 min. with Ac₂O), m. 271-2°, diacetyl derivative (refluxing 3 hrs. with Ac₂O), m. 224.5-6.0°. When heated with BzH 0.5 hrs. at 150-60°, VII formed an anil, m. 239-40° (alc.). (19 g.) added all at once to 50 ml. 85% H₂SO₄ at 100° caused a violent reaction accompanied by charring and evolution of SO₂; the temperature rose rapidly to 170°. The mixture extracted with Et₂O, washed, and dried gave 3 g. BzOH, m. 119-20°. I (5 g.) refluxed 53 hrs. with 400 ml. dilute H₂SO₄ (1:4) and cooled overnight gave 4.3 g. C13H18NO(CO₂H) (VIII), m. 170-2° (aqueous alc.); Me ester (IX), m. 94-5° (aqueous MeOH). VIII (5 g.), 8 ml. 30% H₂O₂, and 50 ml. AcOH heated 5 hrs., diluted with 50 ml. H₂O, and the acid removed in vacuo gave 2.1 g. C13H18NO(CO₂H) (X), m. 219-21° (MeOH). LiAlH₄ (3 g.) and 150 ml. Et₂O refluxed 5 hrs. with 15 g. VIII in 1 l. dry Et₂O, the complex decomposed, and the residue recrystd. gave 2.3 g. C13H20NO(Me), m. 140-1° (C₆H₆-ligroine). VIII (25 g.) and 20 g. Zn dust heated to 250-60° gave vigorous bubbling and at 275° some sublimation. The pressure was reduced to 24 mm., but the material would not distill at a bath temperature of 340°. The charred, tarry material extracted with Me₂CO, the Me₂CO evaporated, and the residue recrystd. gave C13H18NO(CH₂OH) (XI), m. 232-4° (95% alc.). Hydrogenation of 6 g. X in 100 ml. methylcyclohexane over 20 g. Cu chromium oxide catalyst at 220-35° gave 4 g. C13H22NO(Me)

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 AB (Ph₃P)ZnI₂(CN)₄ (I) (gray-green), (Ph₃P)ZnI₃(Cu(CN)₄)₂ (gray), (C₅H₅N)ZnI₂(CN)₄ (violet), (C₅H₅N)ZnI₂(CN)₄ (blue-green), (C₅H₅N)ZnI₂(CN)₄ (light green), PhMe₂NCu(CN)₄ (dark blue), PhNH₂Ni₂(CN)₄ (light blue), (Ph₃P)ZnI₂(CN)₄ (red), Et₃NCu(CN)₄ (gray), and (Ph₃P)ZnNi(CN)₄ (pink) (no m. ps. given) are obtained by addition of an aqueous solution of a complex compound of NaCN, KCN, or NH₄CN with Ni, Co, Fe, Cu, or Ag to an alc. solution of NiCl₂ and Ph₃P, pyridine, or C₅H₅N. For example, 65 parts (Ph₃P)ZnI₂ was dissolved in 500 parts MeOH with stirring and heating to 40-50°. To this solution, 24 parts K₂Ni(CN)₄ in the form of its 14% aqueous solution was added during 24 min., forming a difficultly soluble compound. The suspension was heated for a short time to boiling; the crystalline precipitate sucked off while hot, washed with cold H₂O, and dried at 150° to give a quant. yield of I, insol. in H₂O and most organic solvents and not decomposed or dissolved by NH₄OH. These compds. are useful as catalysts for organic reactions.

ACCESSION NUMBER: 1958:62695 CAPLUS
 DOCUMENT NUMBER: 52:62695
 ORIGINAL REFERENCE NO.: 52:11320e-h
 TITLE: Complex compounds of copper, nickel, and zinc
 INVENTOR(S): Schueckendiek, Walter; Sepp, Karl
 PATENT ASSIGNER(S): Badische Anilin- & Soda-Fabrik AG
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 888849		19530903	DE	

L20 ANSWER 205 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The electrochem. reduction of the o-nitroaniline (I) into o-phenylenediamine (II) successfully proceeds in the aqueous alc. solution of NaOH on the cathodes Hg, Cu, Zn, Al, graphite, Sn, Pb, Fe, Ni, and Pt. The maximum current efficiency of the II was obtained on the cathodes Pb (83%), Hg (79.8%), and Cu (74.5%). The effective electroreduction of I on the Hg cathode can be explained by the high overvoltage of H and on the Cu, by the catalytic action of Cu.
 ACCESSION NUMBER: 1958:14688 CAPLUS
 DOCUMENT NUMBER: 52:14688
 ORIGINAL REFERENCE NO.: 52:2613g-i
 TITLE: Electroreduction of the o-phenylenediamine. I. Effect of cathode material on the electroreduction of o-nitroaniline
 AUTHOR(S): Ter-Minasyan, L. E.
 SOURCE: Izvest. Akad. Nauk Armyan. S.S.R., Ser., Khim. Nauk (1957), 10, 173-80
 DOCUMENT TYPE: Journal
 LANGUAGE: Russian

L20 ANSWER 206 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB cf. C.A. 51, 28261. The Zn-dust dry distillation and LiAlH₄ reductions of tuberostemonine (I) were studied. The catalytic reduction of the dry distillation product of I in EtOH with Pd-black catalyst yields no basic substance. Pressurized hydrogenation in C₆H₆ with Pd-C catalyst also resulted in failure. Attempts to convert the amorphous substance with pos. pyrrole reaction to a crystalline salt also failed. This pyrrole-like substance (0.3 g.) submitted to Adkins' high-pressure reduction gave only 20 mg. basic substance. A Cu-Cr oxide catalyst was used in EtOH, and the mixture heated 3.5 hrs. at 150-60° and an initial pressure of 137-8 atmospheric; no basic substance was obtained. Reaction 3 hrs. at 200° and 160 atmospheric gave some pale yellow, sirupy base which did not crystallize. Its ultraviolet spectrum did not exhibit any characteristic absorption. Thus, it is assumed that, since I is easily dehydrogenated by oxidation with Ag₂O and KMnO₄ to a substance with a pos. pyrrole reaction, it also undergoes dehydrogenation by Zn-dust dry distillation and changes from the pyrrolidine to a pyrrole skeleton which is resistant to reduction. I does not possess an active CO group but is thought to have an inactive CO group; treatment of I with LiAlH₄ in an attempt to reduce the lactone ring yielded an amorphous base (II) giving a pos. Liebermann nitrosamine reaction and a small amount of a neutral substance giving a pos. Ehrlich pyrrole test. Saponification of II with alc. KOH revealed the absence of a lactone ring. Addition of application of MeI could not be effected by warming or heating. Its ultraviolet spectrum did not exhibit any characteristic absorption.
 ACCESSION NUMBER: 1957:90792 CAPLUS
 DOCUMENT NUMBER: 51:90792
 ORIGINAL REFERENCE NO.: 51:16499e-h
 TITLE: Stemon alkaloid. XX. Tuberostemonine. 9
 AUTHOR(S): Kondo, Heisaburo; Satomi, Masakichi; Kaneko, Tsutomu
 CORPORATE SOURCE: ITSUU Lab., Tokyo
 SOURCE: Ann. Rept. ITSUU Lab. (Tokyo) (1957), 8, 15-17
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 207 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Dicyanoketene acetals [prepared from (C(CN)₂)₂ and alcs. in the presence of catalysts, e.g. urea, a tertiary amine, or metal salt] give with 2 mol NaCH(CN) 2 (I) (NC)₂C=C(CH(CN))₂ 2 (II) and salts according to the equation: (NC)₂C=C(2R) 2 + 2NaCH(CN) 2 + (NC)₂C=C(C(CN))₂ 2--2Na+ + 2R₂CH, where 2 is O or S. Thus, dicyanoketene ethylene acetal 68 in hot EtOH 197 parts added to I gave 70% crude II Na salt (24% purity), white granules from Et₂O; the di-Et acetal gave the same product. The Ba salt was obtained as white needles, forming a colorless crystalline hexahydrate, convertible to a trihydrate by vacuum evaporation of a MeOH solution. II was freed from the Ba salt by addition of H₂SO₄ and titrated with NaOH; the free acid is stronger than aqueous H₂SO₄ of the same normality; the second pKa is 2.25 (first not measurable). The following salts were prepared from the Ba salt by addition of the corresponding sulfate:
 Al. 2.5H₂O, Hg. -6.5H₂O, Ag, Cd. 2H₂O, UO₂ 2.2H₂O, Fe++ 2.5H₂O, VO++ 3.5H₂O, Cu++ 0.5H₂O, Ni++ 4H₂O, Co++ 2H₂O, Sr. 7H₂O, Zn. 2.5H₂O, Mn++ 2H₂O, Hg++ 4H₂O, and C₆H₅NH₃. II 20.47 in H₂O 1000 and quinoline 50 in 54 HCl 1025 parts gave the diquinolinium salt of II, light yellow needles, m. 206-8°. The Na salt of II and Pr₄N⁺ gave the di-Pr₄N salt of II, m. 296-8°; also Bu₄N⁺ gave the analogous Bu₄N salt. Thioacetals may be used without disadvantage. Temps. of 0-50° may be used. The Ba, Zn, Sr, and K salts are luminescent in UV and cathode rays and may be useful as phosphors in television tubes. Cf. preceding and following abstract
 ACCESSION NUMBER: 1957:62432 CAPLUS
 DOCUMENT NUMBER: 51:62432
 ORIGINAL REFERENCE NO.: 51:11372e-i
 TITLE: 2-Dicyanomethylene-1,1,3,3-tetracyanopropane and its salts
 INVENTOR(S): Middleton, Wm. J.
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2766246		19561009	US	

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 GI For diagram(s), see printed CA Issue.
 AB CH₂:CHOR (I) could be prepared by any of the following methods, besides the use of CH₂:tpbond.CH (II): ClCH₂CH(OR) 2 + Na or Mg; ICH₂CH₂OR + EtONa; MeCH(OR) 2 with loss of ROH; MeCHCl₂ + 2RONa; (CH₂Cl)₂ + 2RONa; CH₂:CHCl (III) + or R₂ONa. The last of these reactions is not in harmony with literature statements that point to the sluggish behavior of halogen derivs. of type III, which in the presence of alcoholates are claimed to give, preferentially, II and HCl. It was found that in this reaction the yields of II were normally very low, especially when the mixture was kept some time after the reaction appeared to be complete. Any II that is formed from III may lead to I, in the following manner: II + ROH → (ROCH:CHK) + ROH + I + ROK. Kinetic studies indicate that the addition of RO- to II is the step that most fully detes. the rate of I formation. Vinylation of various alcs. is catalyzed in the liquid phase by 0.5-1% alkali metals, 1-2% hydroxides (especially KOH), and KCN. In vinylating in the gaseous phase, the alc-II mixture is passed over alkali (e.g., soda-lime) at 150-350°. Often the vinylation temps. are 150-80°; MeOH requires about 120°. Primary and sec. alcs. can be vinylated readily to form I; tertiary alcs. react more slowly. HOCH₂(CH₂)_nCH₂OH (n = 0, 1, or 2) with II may yield (CH₂:CHOCCH₂)₂(CH₂)_nCH₂:CHOCCH₂(CH₂)_nCH₂OH, or, at higher temps., MeCH.O.CH₂(CH₂)_n.CH₂O. At 200°, (CH₂OH)₂ and II gave exclusively MeCH.O.CH₂.CH₂O (IV). Conditions are also given briefly for the vinylation of sugar acetone derivs. Vinylation of free sugars proved unsatisfactory. Phenols and naphthols usually vinylate sluggishly, but in methylpyrrolidone, using K, they react readily and quant. with II to form aryl vinyl ethers. Using Zn or Cd salts, or organic bases as catalysts, the phenols normally polymerized to form resins. Certain polysubstituted phenols (e.g., 2,4,6-Cl₃CGH₂OH) gave vinyl ethers that failed to polymerize. Hydroxystyrenes when vinylated gave resins. The reactivities of substituted phenols towards II were in the following order m > p > o. RN'NCH₂CH₂OH with II gave RN'N(CH₂)₂CH₂CH₂. Hydroxysterilamines [e.g., PhNH(CH₂)₂OH] with II and KOH formed vinyl ethers that cyclized readily, especially with Zn(OAc)₂, to form the corresponding aryl-2-Me oxazolidines. Typical reactions of I were given. With NH₃ and suitable catalysts at 200-250°, I gave largely 2-methyl-5-ethylpyridine. Conditions for the polymerization of I are discussed, including the reversible "coagulation point" of aqueous solns. of [H₂COCH(OR)]_n which flocculate above 35°, and the precipitate from which redissolves below 35°. Precautions are given in the use of II. In the formation of the following I, KOH was the usual catalyst, although alcoholates and other catalysts were sometimes used. In a number of instances, other methods were used in forming I, from halogenated products or acetals, as outlined above. Pressures varied from 15-30 atmospheric and temps. from 120-180°. ROCH:CH₂ were formed from the appropriate alc. [R and the b.p. (at 760 mm. unless otherwise stated) (the designation (a) indicates that anal. data are given) given]: Me (a), 8°; Et (a), 36°; Pr, 65°; Bu (a), 93°; iso-Bu (a), 82-3°; Me₂CHCH₂Me, 109-112°; n-octyl, 75°/5 mm.; 2-ethylhexyl, 74-6°/12 mm.; n-octadecyl (a), 174°/1 mm. (m. 29°), hydrogenated in MeOH with Ni, giving EtOC₁₈H₃₇, m. 34°, b₁₀ 190°; iso-Pr, 55°; sec-Bu, 30-33°/55 mm.; Am, 111°; iso-Am, 112°; isohexyl, 40-2°/14 mm.; isooctyl, 50-55°/14 mm.; 2-methyl-n-octyl, 72-5°/12 mm.; n-decyl, 120°/12 mm.; 6-undecyl,

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 82-5°/2 mm.; n-dodecyl, 120°/4 mm.; n-tetradecyl, 142°/2 mm.; n-hexadecyl, 160°/2 mm.; oleyl, 170-80°/1 mm. EtCH(OEt)2 passed over 10A Asbestos at 280° gave 75% MeCH:CHOEt, b. 69°. Me2C(OEt)2 gave 60% MeCH:CHOEt, b. 62° (also formed from MeCH:CH2 and EtONa in abs. EtOH at 100°, and from MeCH:CHCl and EtONa at 180°, either case together with very small amts. of MeCl and EtOH). PrCH(OEt)2 passed over an Ag-kieselguhr catalyst gave 75% MeCH:CH2:CHOMe, b. 73-4°. EtONa in abs. EtOH and MeCH:CHCl or MeCH:CH2 at 180° gave MeCH:CHOEt, b. 70-72°. UO2(NO3)2 (15 g.) and 450 g. clay shards after heating at 400°, cooling, treatment with 45 g. Ag2CO3 in NH4OH, and heating in H at 270° gave a catalyst which at 220° converted a stream of PhOMe(OEt)2 to 85% PhC(OEt):CH2, b12.5 93-6°. The vinylation of 1 kg. (CH2OH)2 contg. 5 g. KOH at 120° gave 60 g. IV, b. 82°, 180 g. (CH2OCH:CH2)2, b. 124°, and 460 g. HOCH2CH2OCH:CH2, b. 135-40°; 500 g. glycol was recovered. At higher temps. IV was the main product. The following were also prepd. but no details are given: CH2:CHOCCH2CH2OCH2CH2OH, b12 108°; (CH2:CHOCCH2CH2)2O, b12 85°; and (CH2:CHOCCH2CH2OCH2)2, b12 90-96°. EtOCH2CH2OCH(OH)1 kg.), KOH, and II at 150° gave 1.1 kg. EtOCH2CH2OCH:CH2 (VI), b. 126°, also formed in 60% yield by passing MeCH(OCH2CH2OEt)2 over Pt-asbestos at 260°. V, hydrogenated with Ni as catalyst gave EtOCH2CH2OCH2CH2, b. 108-9°. CH2:CHOCCH2CH2OCH2CH2, b2 61°. AcOCH2CH2OH (104 g.) heated with II and 20 g. Zn α -methylvalerate gave 56 g. CH2:CHOCCH2CH2OAc, b0.2 40-1°. The following ROCH2CH2CH:CH2 were formed by treating ROCH2CH2OH with II (R, b.p.s., and m.p.s. (in parentheses) given): Ph, 122°/16, (26°); p-ClC6H4, 113-15°/2; p-MeC6H4, 133°/16; p-xylol, 126-7°/10; p-BuC6H4, 137-9°/15; p-iso-BuC6H4, 130°/4; p-tert-BuC6H4, 126-7°/10; 2,5-(iso-Bu)2C6H3, 156-9°/1; p-n-dodecylphenyl, 195-8°/4; p-n-octadecylphenyl, 208-12°/0.6; β -naphthyl, 158-60°/2, (65°); 1,3-(CH2:CHOCCH2CH2)2C6H4, b2 216-20°. Vinylation of MeOCH2CH2OH with II and Cd(OAc)2 catalyst gave 250 g. CH2:CHOCCH2CH2OMe, b18 78-80°. EtOCH2CH2OCH2CH2OCH2CH2, b16 90-2°; EtO(CH2)2O(CH2)2OCH2CH2CH2, b10 110-20°; (CH2CH2OCH2CH2)2, 60-5°; EtO(CH2)2OCH:CH2, b3 55°; 1,12-octadecanediol divinyl ether, b2 203-5°; a monovinyl ether of HOCH2CH2CH2OMe(OH), b. 117-20°. Vinylation of 1 kg. glycerol using 10 g. KOH at 160-80° gave 250 g. of org. phase, either a monovinyl ether or the isomeric MeCH:OCH2CH:CH(CH2OH)O, (a), b2 65-7°, also formed in higher yield from glycerol, KOH, and CH2:CHBr at 200-220°. The following 1,2-O-derivs. of HOCH2CH(OH)CH2OCH:CH2 were formed: CH2:2, b10 53°; MeCH:2, b9 56°; Me2C:2, b10 57°; PhCH:2, b2 120°. From glycerol diacetate, II, and Zn naphthenate at 170° were formed a somewhat impure divinyl ether of monoacetylglycerol, b0.4 64-66°, and an impure monovinyl ether of diacetylglycerol, b0.2 72-80°. Isopropylideneglucose with KOH suspended in PhMe took up 1 mol II on shaking giving a pale yellow viscous sirup. β -Diacetonfructose, KOH, and II-N at 150-60° gave a vinyl ether, b3-4 130-34°, m. 43-5°, sol. in EtOH, Me2CO, C6H6 and ligroine. Vinylation of diacetonoglucose gave an impure vinyl ether, b1 104-22°. No analyses are given. PhONa (50 g.), 1.25 l. MeOH, and 470 g. PhOH with II-N at 180° gave 480 g. PhOCH:CH2 (a), b17 54-5°, also formed by passing 211 PhOH-II over Zn(OAc)2 or Cd(OAc)2 at 270°. α -MeC6H4ONa in EtOH with CH:CHCl at 180° gave in good yield, after suitable fractionation, and Et2O extrn., α -MeC6H4OCH:CH2 (a),

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 155-60°. 3-Hydroxytetrahydrofuran (120 g.) subjected to the usual vinylation gave 110 g. vinyl 3-tetrahydrofuryl ether (XI), b18 49°, yielding 3-ethoxytetrahydrofuran on hydrogenation in cyclohexane. Prepd. from appropriate THF derivs. were the 2,5-di-Me deriv. of XI, b18 64-6°, and the 4-MeO deriv. of XI, b34 82-3°. Tetrahydrofurfuryl alc. (1 kg.) on vinylation using 10 g. Na gave 1.05 kg. vinyl tetrahydrofuryl ether, b13 56-8°, which gave the corresponding Et ether, b. 149-51°, on hydrogenation. By various of the reactions outlined above, the following derivs. of the appropriate compounds, I, were prepd. With dry HCl, I (R = Et) (Ia) at 0° gave MeCHClOEt (XII), b. 92-5°. I (R = Bu) (Ib) gave MeCHClOBu (XIII), b14 38°, decomp. when distd. at 760 mm.; MeCHClOPh, b12 79-81°. Ia added dropwise to BuOH and a few drops of H2SO4 below 30° gave MeCH(OBu)OEt (a), b. 151-2°, also formed from Ib and EtOH, or from XIII and EtONa. Ia and EtOH gave MeCH(OEt)2, b. 102°; BuOH and Ib gave MeCH(OBu)2, b. 183-4°. To HOCH2CH2Cl (80.5 g.) in 70 cc. Et2O contg. 4 drops of SOCl2 was added slowly at 15° 75 g. Ia followed by 10A NaOH, and the org. phase sepd. to yield 76 g. MeCH(OEt)OCH2CH2Cl (a), b10 54-5°, higher reaction temps. gave mixts. and lowered the yield. Ia and XII in Et2O with (CH2OH)2 at 30° gave [MeCH(OEt)OCH2]2 (a), b18 97°. Ia (80 g.) in dry Et2O contg. small amts. of XII with 90 g. PhOH gave, after extrn. with NaOH, and sepn. of the org. phase, 140 g. MeCH(OEt)OPh (a), b10.5 83-4°, b. 200-2°. With slight modifications the following acetals were formed: (from Ia and VII) MeCH(OEt)OC6H11, b10 72-3°, with small amts. of MeCH(OEt)2 and MeCH(OC6H11)2, b11 136°; from BuOH and I (R = CH2Ph) contg. traces of BF3, MeCH(OBu)OCH2Ph, b17 135-8°, also formed from Ib and PhCH2OH; from Ia and MeCH(OH)CO2Et, MeCH(OEt)OCH2MeCO2Et, b10 76°; from EtOH and MeCH:CHOEt with fuller's earth EtCH(OEt)2, b. 124°; from MeOH and CH2:CHMeOEt, Me2C(OMe)OEt, b. 96°. Ia added gradually to glacial AcOH contg. a few drops of H2SO4 at 20°, followed by Na2CO3, gave AcOCH2MeCO2Et, b. 128-30°. HOCH2CHMeOEt, b. 73-4°. Ib and AcOH contg. fuller's earth, or XIII and AcOH, gave AcOCH2MeOBu, b. 163°. Ib and PrCO2H formed PrCO2CHMeOBu, b10 78-80°. From 60 g. powd. α -HOCH2CH2CO2H, 40 g. Ia, and 3 g. XII at 55° with stirring, and after Et2O extrn. and shaking with 3% NaOH, was formed α -ethoxyethyl salicylate (a), b0.025 84-5°. Ib and XIII yielded α -butoxyethyl salicylate, b0.006 92-3°. To 18 g. AcOH and 3 g. dry NaHSO4 stirred at 20-25° was added dropwise EtCH:CHOMe followed by Et2O and neutralization with Na2CO3; the dried Et2O phase gave 30 g. PrCH(OMe)CH2OAc, b16.5 55-6°. I (R = Me) (29 g.) shaken 6 h. at 200° with 13 g. HCN and 50 cc. pyridine, distd., neutralized with HCl, extrd. with Et2O, and redistd. gave 2 g. MeCH(OMe)CN (a), b. 118°. MeCH(OEt)CN (a) (XIV), b. 130°; MeCH(OBu)CN (a), b20 70°; MeCH(OBu-iso)CN, b33 72°; α -cyclohexyloxypropionitrile (a), b0.5 85°; α -(2-decahydronaphthyl)oxypropionitrile (a), C13H21ON, b0.5 102-3°. Sapon. of XIV and acidification gave MeCH(OEt)CO2H, b16 100°. MeCH(OBu)CO2H (a) b0.5 77-8°. Hydrogenation of 200 g. XIV with 40 g. Raney Ni and 40 g. NH4OH at 225° and 200 atm. gave 139 g. MeCH(OEt)CH2NH2 (a), b. 118°; and 48 g. MeCH(OEt)CH2)2NH2, b37 111-12°. MeCH(OBu)CH2NH2 b35 69°. Ia (79 g.) in 34 g. liq. NH3 and 40 cc. EtOH was shaken and heated to 250° with 1 g. CuCl and 2 g. NH4Cl giving 24 g. 2-methyl-5-ethylpyridine (XV) (a), b18 71-2°; picrate, m. 164°. Other Cu or Fe catalysts could replace CuCl. When treated similarly other homologs of Ia also gave XV. Ia in NH4OH heated 30 h. with Raney Ni at 260° gave AcNHET (a), b18 104-5°. Valeric acid (290 g.)

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 b. 168-70°. Analogously prepd. were the m-isomer (a), b. 175-77° [also formed from (CH2Cl)2 and m-MeC6H4OK in EtOH], and the p-isomer (a), b18 75°. The following ROCH:CH2 were prepd. (R given): p-tert-BuC6H4 (a), b20 112-13°; p-isobutylphenyl (a) [by vinylation using KOH and II], b1 91-3°; p-iso-octylphenyl (a), b2 107-10°; α -ClC6H4 (a), b25 88°; p-ClC6H4 (a), b18 89°; 2,4-Cl2C6H3, b14 104-5°; 2,4,6-Cl3C6H2 (a), m. 36-8°, b14 117-18°; Cl5C6, m. 91-3°, b1.2 137-9°; PhCH2, b25 88-92°; p-ClC6H4CH2, b10 99-101°; MeCHPhCH2, b10 89-92°; Ph(CH2)3, b12 120-2°; Ph2CH, b15 120°; MeCHPh, b. 224°, b14 70-3°; PhCH2CH2, b20 95-7°. 1,3-(CH2:CHO)2C6H3 (a), b24 104°, and its 1,4-isomer (a), b23 107°. CH2:CHCl and K α -naphthoxide in EtOH gave α -ClCH7OCH:CH2 (a), m. 33°, b18 136°; its β -isomer (VI) (a), m. 35°, b21 147°, the tetrahydro deriv. of VI (a), b18 143°; decahydro deriv. of VI (a), b1 118-21° (on further catalytic hydrogenation gave ClOH17OEt, b20 118-24°). Anthrone (300 g.) in PhMe with Zn stearate, on vinylation with 3:1 II-N at 20-25 atm. gave vinyl anthryl ether, yellow, b4 175-85°, m. 47°. Vinyl cyclohexyl ether, b15 53-5°, was formed either from cyclohexanol (VII) and II at 150° (using KOH) or from VII and KOH, followed by stirring 8 h. with (CH2Br)2 at 130° and steam disty. The following compds. cyclohexyl derivs. of type I were formed (R and b.p. given): p-BuC6H10, 98-102°/4; 1,4-(iso-Bu)2C6H5, 138-40°/2; p-n-dodecylcyclohexyl, 117-21°/4.5; C6H11CH2, 46-7°/5; C6H11CHMe, 68-9°/15; C6H11CH2CH2, 93-5°/25. Decahydronaphthylmethyl vinyl ether, b4 117-21°. The following vinyl ethers were formed by vinylation of terpene alcs. at 180° using BuONa in BuOH (R and b.p. given): carvacryl, 99-102°/16; terpineyl, 78-80°/2; bornyl, 72-4°/2; isobornyl, 66-71°/2; menthyl, 95-6°/17. Dipentenylicarbinol (VIII) was formed by treating dipentene with CO and H, using Co catalysts, followed by subsequent catalytic hydrogenation of the CHO group (catalyst not given). VIII CH2:CH ether b1 79-81°; this in cyclohexane hydrogenated with Ni-Cr2O3 gave VIII Et ether, b1 79-81°, having a hyacinth odor. Formed analogously to VIII was camphaneiccarbinol vinyl ether, b29 122-5°. Vinyl dihydroabietinyl ether b2 178-83°. To Me2CHCH2CH2OH (700 g.) under N at about 110° was added 7 g. Na and the mixt. treated at 130-40° with II-N yielding 600 g. Me2CHCH2CH2OCH:CH2 (IX), b23, 38-41°, after distn. over KOH. The N,N-di-Et homolog of IX, b12 46-7°; N,N-di-Bu homolog, b10 100-2°; N-mono-Bu analog of IX, b3 70-2°. Et2NCH2CH2CH2CH2OMe(OCH:CH2) b24 112-15°; HOCH2CH2NHCNCH2CH2OCH:CH2, b8 102-6°; (HOCH2CH2)2NHCNCH2CH2OCH:CH2, b8 124-8°. PhNHCH2CH2OCH:CH2 (a), b9-10 128-32° (1.6 kg.), was obtained by vinylation of 1.5 kg. PhNHCH2CH2OH (X), using KOH in BuOH as a catalyst; hydrogenation of the vinyl deriv. in MeOH gave PhNHCH2CH2OEt, b14-15 132-6°. When X in BuOH was heated with Cd(OAc)2 (in place of KOH) in BuOH and II, 1-phenyl-2-methyloxazolidine (a), b. 249°, m. 60°, was formed. Using II, the following I were formed (R and b.p. given): m-MeC6H4NHCH2CH2, 142-5°/11; m-Cl analog, 234-7°/14; Ph2NCH2CH2, 155-60°/3; PhNHCH2CH2, 117-18°/4; PhNBuCH2CH2, 148-51°/10 [hydrogenated gave EtO(CH2)2NBuPh, b12 156-9°]; pyrrolidylethyl, 80°/16; α -pyrrolidonyl ethyl, 128-33°/3; hexamethyleniminoethyl, 103-5°/15. PhNHCH2CH2OCH:CH2)2, b3 142-3°. m-ClC6H4N(CH2CH2OCH:CH2)2, b12 198-20° (hydrogenated to the di-Et ether, b12 187-9°). Perhydrocarbazolyethyl vinyl ether b3

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 contg. 5.8 g. ZnO in 350 cc. PhMe at 180° was treated with I-N 20 h. at 20-25 atm., cooled, filtered and distd. giving 300 g. vinyl valerate, b. 134°. Formed similarly were the vinyl esters of the following acids: caprylic, b12 94°; 2-ethylcaproic, b20 128-30°; lauric, b14 123°; myristic, b3 150°; stearic, b2 178°; oleic, b2 175°; palmitic, b2 165°. Using CdO and the appropriate acid dild. with PhMe and vinylating with II-N at 180-85° yielded vinyl esters of the following acids: BzOH, b3 113-15°; PhCH2CO2H, b4 88-90°; PhCH:CHCO2H, b4 133°; 2-AcOC6H4CO2H, b5 147-9°; 2-EtO2CC6H4CO2H, b3 173°; α -naphthoic, b5 147-52°; β -naphthoic, b4 153°; abietic, b2 196-8°. 188 refs.
 ACCESSION NUMBER: 1957:51813 CAPLUS
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 ORIGINAL REFERENCE NO.: 51:9577a-1,9578a-1,9579a-1,9580a-h
 TITLE: Vinylation. I. Vinyl ethers and vinyl esters
 AUTHOR(S): Rapp, Walter; et al.
 CORPORATE SOURCE: Badische Anilin & Soda Fabrik A.-G., Ludwigshafen/Rhine, Germany
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 OTHER SOURCE(S): CASREACT 51:51813

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 AB cf. C.A. 50, 10110b. Aristolochic acid (I), C₁₇H₁₁O₇N, was identified as 3,4-methylenedioxy-8-methoxy-10-nitro-1-phenanthrenecarboxylic acid. I was isolated from the dried root powder of Aristolochia clematitis by degreasing with petr. ether, extracting with EtOH, evaporating the alc. in vacuo, treating the residue with dilute soda solution and ether, after separating the ether solution acidifying the soda solution with HCl to form a brown precipitate, boiling the crude acid precipitate under reflux 3 times with a little alc., digesting repeatedly with dilute KMnO₄ solution until no more dissolves, acidifying these solns. individually, combining the ppts. from all but the first, and recrystg. from HCONHMe₂-alc. (1:6) to crystalline I, dried at 140° in vacuo, m. 281-6° (decomposition). I was esterified with CH₂N₂ in dioxane to its Me ester (II), m. 281°, and decarboxylated with Cu powder in quinoline to 744 compound (III), C₁₆H₁₁O₅N, m. 212° (3,4-methylenedioxy-8-methoxy-10-nitrophenanthrene). Hydrogenation of both I and II in AcOH with a Pt catalyst gave a compound, C₁₇H₁₁O₄N, m. 319° (3,4-methylenedioxy-8-methoxy-10-amino-1-phenanthrenecarboxylic acid lactam). Hydrogenation of III in alc. with Pd-C as catalyst yielded a compound, C₁₆H₁₃O₃N, m. 170° (3,4-methylenedioxy-8-methoxy-10-amino-1-phenanthrene). In Ac₂O with NaOAc and in dust, III yielded a compound (IV), C₁₈H₁₅O₄N, m. 274° (3,4-methylenedioxy-8-methoxy-10-acetamidophenanthrene). Zinc dust distillation of I gave phenanthrene. Oxidative destruction of II in alkaline tetrahydrofuran with H₂O₂ yielded a compound (V). C₁₆H₁₂O₇, m. 243° (5,6-methylenedioxy-3'-methoxy-2,2'-biphenyldicarboxylic acid), which on methylation with CH₂N₂ in MeOH gave the di-Me ester, m. 114°. Ether splitting from V in resorcinol with concentrated HCl in a bomb tube (3 hrs. at 130°) yielded 654 of a compound (VI), C₁₅H₈O₄, m. 204° (2,3,3'-trihydroxy-2'-biphenylcarboxylic acid lactone), which methylated with CH₂N₂ in MeOH gave the di-Me ether (VII), m. 198°. VII was oxidized with excess KMnO₄ at pH 8 to o-methoxyphthalic anhydride, m. 160°. 1,5,6-Trimethoxy-10-phenanthrenecarboxylic acid in AcOH with Na₂Cr₂O₇ gave 604 1,5,6-trimethoxy-9,10-phenanthrenequinone, m. 167°, which decomposed in alkaline MeOH solution with H₂O₂ gave 3',5,6-trimethoxy-2,2'-biphenyldicarboxylic acid; this on treatment with concentrated HCl in a bomb tube (3 hrs. at 130°) gave a compound, which on admixt. with alc. showed no m.p. depression. The di-Me ether of the synthetic lactone was identical with VII. Determination of methoxyl groups was carried out in the apparatus of Elek (C.A. 33, 28452) by a modified method. Samples of 3-5 mg. dissolved in 0.3 ml. (EtCO)₂O were treated after cooling with 2 ml. constant-boiling HI and 0.5 ml. HI of d. 1.96, and heated 45-60 min. under the usual conditions. Ultraviolet absorption spectra are included for III in EtOH (compared with 9-nitrophenanthrene) and IV in EtOH (compared with 9-acetamidophenanthrene) and infrared spectra for solid I in KBr and III and IV in Nujol. 44 references.

ACCESSION NUMBER: 1957:5407 CAPLUS
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 AB XIIa is prepared in quant. yield from 1 kg. (HOCH₂CH₂)₂ stirred and heated to 165° with 100 g. 80% H₃PO₄ with fresh IX added and the temperature raised slowly to 185° as product distilled, and the distillate dried over K₂CO₃ and redistd., b. 65-7°, d₂₀ 0.8886, n_D20 1.4065; azeotrope containing 5.5% H₂O b. 66°. XIIa is also prepared from IX with p-MeC₆H₄SO₃H, (COCH₂)₂, (CH₂COCH₂)₂, maleic anhydride, or resorcinol-(HO₃S)C₆H₃CHOCH₂O resin as catalyst, by heating 35% aqueous IX with H₃PO₄ and NH₄Cl in an autoclave to 280° 10 h., or from 25 g. IX passed over 325 g. precipitated and calcined Cr₂O₃ at 300-25° or CaHPO₄ at 300°. Dehydration of the corresponding R₁R₂C(OH)(CH₂CH₂CH₂CH₂OH)R₃R₄ gives the following cyclic ethers (R₁, R₂, R₃, R₄, and catalyst given): Me, H, H, H (XXXV), H₃PO₄-H₂SO₄, 78-9°; Me, H, Me, H, K₂S₂O₇, 91.5° (azeotrope containing 13% H₂O, b. 78°), forms with ferrocyanic acid an H₂O-insol., colorless, crystalline adduct; Me, Me, Me, H₃PO₄ or K₂S₂O₇, b. 114-17°. 1,1'-Ethylenedicyclohexanol (120 g.) gives, with 2 ml. 25% H₂SO₄ in vacuo at 150°, 85-g. 2,2,5,5-bis(pentamethylene)-XIIa, b₂ 93-5°. (CH₂CH₂)₂ is prepared by passing 20-5 g. IX and 4-5 g. H₂O/h. at 300-50° over 300 mL catalyst prepared by mixing 100 g. anhydrous NaH₂PO₄ with 40 mL H₂O, adding 8 g. BuNH₂PO₄ and 20 g. graphite, evaporating with continuous stirring, heating finally to 160°, and breaking to suitable size. The same catalyst, with 98% H₃PO₄ instead of Bu phosphate, gave 90% (CH₂CH₂)₂ from XIIa at 280°; 1800 g. XXXV gives 510 g. MeCH₂CHCH₂CH₂, b. 40°, and 720 g. recovered XXXV. [MeCH(OH)CH₂]₂ (500 g./day), passed at 280-300° over catalyst prepared by adding 125 g. Al(OH)₃ to 700 mL H₂O and 700 g. 90% H₃PO₄, stirring 2-3 h. at 110-15°, adding 185 g. NaH₂PO₄ and 43 g. BuNH₂, cooling, adding 320 g. 34.4% water glass, ball milling, evaporating in vacuo at 260°, and crushing, gives 240 g. (MeCH₂CH₂)₂, b. 77-8°. 2,5-Dihydrofuran (17 g.), prepared from 50 g. XXV and 2 g. 20% H₂SO₄ at 125-30° and 18 mm., b. 63.5° (forms azeotrope containing 7.5% H₂O). XXV is also dehydrated by (COCH₂)₂ at 170°, by passing in HCl at 140° or by passing it over Al₂O₃ at 240-50°. 2,5-di-Me-XXXVa, b. 90-3°, and 2,2,5,5-bis(pentamethylene)dihydrofuran, b₁₆ 123-5°, are prepared similarly. 1,2,4-Butanetriol (300 g.) and 10 mL concentrated H₂SO₄ at 30 and 100-15° treated with an addnl. 1500 g. triol and distilled during 36 h. give 1350 g. 3-HO-XIIa (XXXVII), b₇₄₀ 183°, b₂₀ 93-5°, acetate, b₁₂ 64°. XXXVI (88 g.) added at -5° to 108 g. COCl₂, warmed to 20° in 1 h. and swept with dry air, the residual chloroformate (XXXVII) treated with 100 mL NH₄OH at 20-40°, then a stream of NH₃ gas, and extracted with Et₂O, and the extract evaporated gives 100 g. XXXVI urethane (C₅H₉NO₃), m. 91° (from C₆H₆). PhNH₂ and XXXVII give the N-Ph urethane, m. 112°, and H₂N(CH₂)₆NH₂ gives N,N'-hexamethylenbis(tetrahydro-3-furyl urethane), m. 113°. XXXVI (176 g.), 1 g. Na, and 125 g. CH₂CHCN give B-(tetrahydro-3-furyloxy)propionitrile, b₂ 100-105°. HOCH₂CH₂CH₂CH₂OH and concentrated H₂SO₄ at 150° give 1-methyl-4-hydroxy-XIIa, b. 183°, b₂₀ 90-1°. 2,3-Dichloro-XIIa (XXXVIII), b₂₀ 62° (1200 g.) is prepared by passing Cl into 1500 g. XIIa at 0-10° 60 h.; 500 g. 2-(4-chlorobutoxy)-3-chloro-XIIa (XXXIX), b₂₀ 145-55°, is also formed. XXXVIII (75%) and XXXIX (25%) are also prepared by passing XIIa and Cl diluted with N over glass beads at 100°; XXXIX is prepared in quant. yield by passing HCl into 156 g. XIIa and 280 g. XXXVIII at 20-30° (or in 330 g. yield from 200 g. XXXVIII and 180 g. Cl(CH₂)₄OH 2 h. at 100°). XXXVIII (1 kg.) chlorinated several days at 100°

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 gives 890 g. b₂₇₋₃₀ 120-4°, 1200 g. b₂₈ 140-2°, and hexachloro XIIa isolated from the mixt. m. 40°. XXXVIII (300 g.), warmed with 500 mL H₂O and 250 g. CaO₃ gives 97 g. 2-hydroxy-3-chloro-XIIa (YOH) (XL), b₁₃ 92-5°, and 32 g. Y₂O, b₀ 6.106°. The following ethers are prepd. from XXXVIII and ROH at 100°: YOMe, b. 150°, b₁₄ 50-1°; YOEt, b₁₇ 60-2°; YOBu, b₁₅ 86-8°; YOCH₂CH₂-n, b₂ 166-7°; YOCH₂CH₂OH, b₁₂ 127-30°; (YOCH₂)₂, b₁ 140-5°; the IX YOCH₂C.tpbond.CH₂OH, b₀ 5.135°; (YOCH₂C.tpbond.)₂, b₀ 8.168-9°; YOCH(CH₂OY)₂, b₀ 4.205°; YOCH₂CH(OY)CH₂CH₂OY, b₄ 150-80°. Other YR prepd. from XXXVIII (R, yield, wt. XXXVIII, wt. other reagent, and conditions given): AcO (b₂₅ 110-15°), 200 g., 420 g., 181 g. AcOH, refluxed 1 h.; NaO₃S, 380 g., 280 g., 500 g. cryst. Na₂SO₃ in H₂O; cyano (b₂₁ 97-9°), -, 140 g. and 120 g. CuCN at 100° [concd. H₂SO₄ give the acid, m. 91° (Et ester, b₁₇ 100-6°)]; Ph (b₁₀ 123-32°), 154 g., 280 g. 1 l. C₆H₆ treated several hrs. at 5-10° with BF₃; 1-naphthyl (b₀ 5.171-3°), 1 l. C₆H₆ treated several hrs. with AlCl₃. XXXVIII (141 g.), and 76 g. (NH₂)₂C₆S in 400 mL H₂O refluxed 4 h. and neutralized with 1100 g. 40% NaOH give 110 g. 2-amino-5-(2-hydroxyethyl)thiazole, m. 98.5° (from C₆H₆); mono-HCl salt, m. 227°; mono-Ac deriv., m. 209°, di-Ac deriv., m. 163-4°. Urea gives a low yield of 2-amino-5-(2-hydroxyethyl)oxazole, m. 132-3° (from alc.); mono-HCl salt, m. 207° (from AcOH). XIIa satd. in the cold with HCl every 12 h. for 48 h., shaken with H₂O, and neutralized, gives HO(CH₂)₄Cl, b₁₅ 76-8°, also prepd. from (CH₂CH₂CH₂CH₂)₂ and HCl at 80° with 5% BiCl₃-SiO₂. Cl(CH₂)₄Cl (XLI), b₁₉ 55-6° (560 g.), is prepd. from 360 g. XIIa, 10 g. anhyd. ZnCl₂, and 650 g. SOCl₂ at 130°; it is also prepd. from XIIa and anhyd. HCl 5 h. at 150° or from XIIa, concd. H₂SO₄, and concd. HCl in 4 h. at 170°. XIIa (870 g.), 60 g. concd. H₂SO₄, and 750 g. SOCl₂ kept 15 h. at 65-85° and another 100 g. SOCl₂ added at 100-10° give 970 g. O(CH₂)₄Cl₂, b₁₂ 125-8°. A similar reaction at 100°, 72 h. gives some [Cl(CH₂)₄OC(CH₂)₂]₂, b₀ 6144-7°. The following comds. are prepd. in generally lower yield from the corresponding monosubstituted XIIa by reactions analogous to the foregoing: ClCH₂CH(OEt)CH₂CH₂Cl, b₈ 65-80°, and 4,4'-dichlorodithiodibutyl ether, b₈ 10.140-5°; ClCH₂CH(OMe)CH₂CH₂Cl, b₈ 5.67°, and 4,4'-dichlorodimethoxydibutyl ether; Cl(CH₂)₃CHClMe, b₁₁ 53-4°, and (MeCHClCH₂CH₂CH₂)₂, b₁₀ 125-40°; ClCH₂CHMeCH₂CH₂Cl, b₈ 9.46-66°, and 4,4'-dichlorodimethyldibutyl ether, b₈ 10.125-38°. Similarly XXI gives (MeCHClCH₂)₂, b. 170-2°. Tetrahydrofuran (172 g.), 20 g. anhyd. ZnCl₂, and 300 g. SOCl₂ refluxed 42 h. give 167 g. Cl(CH₂)₅Cl, b₁₆ 75-6°. XXXVa (280 g.), 15 g. anhyd. ZnCl₂, and 540 g. SOCl₂ 7 h. at 80-5° give 100 g. (ClCH₂CH₂)₂ (XLI), b₁₁ 48-51°, b. 145°, better prepd. (410-g. yield) from 320 g. XXV refluxed with an equal amt. of (CH₂Cl)₂ or C₆H₆ in an HCl stream with azeotropic distn. of H₂O; at 0°, mostly ClCH₂CH₂CH₂CH₂OH, b₁₀ 75-6°, is formed. Substitution of HBr for HCl in the corresponding reaction gives Br(CH₂)₄Br, b₂₃ 90-3°; Br(CH₂)₅Br, b₁₈ 104-5°, and [BrCH₂CH₂CH₂CH₂]₂, b₁₄ 74-6°, m. 54°. XIIa (72 g.), 148 g. BuOH, and 13 g. concd. H₂SO₄ heated 5 h. at 155° give 46 g. (BuOCH₂CH₂)₂, b₁₆ 116-20°. XIIa (148 g.), contg. some Zn, treated with 158 g. AcCl gives 500 g. Cl(CH₂)₄OC₂H₅ and 19 g. 90-2°. Other Cl(CH₂)₄OC₂CR prepd. analogously, using Zn or ZnCl₂ are (R and consts. given): ClCH₂, b₁₇ 124°; Pr, b₂₁ 116-18°; Ph, b₂₀ 176-8°; p-O₂NC₆H₄, b₀ 7.167-72°, m. 38°; p-HSO₃CH₂CH₂, b₆ 90-5°; Cl, b₁₀ 90°; Cl(CH₂)₄OC₂CH₂, m. 77° (from aq. alc.); [CH₂NHCO₂(CH₂)₄Cl]₂, m. 104° (from MeOH);

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 [CH2CH2CH2NHCO2(CH2)4Cl]2, m. 94° (from MeOH). o-C6H4(COC1)2 (203 g.), 144 g. X11a, and Zn give 250 g. o-C6H4[CO2(CH2)4Cl]2. Similarly XXXVIII gives ClCH2CH2CH2CH2CH2CO2Ac, b50 154-8°, and XXXV gives MeCHCl(CH2)3CO2Ac, b21 92-5°. XXXV (172 g.) and 230 g. Ac2O heated 12 h. at 240° in a Cu-lined autoclave give 110 g. AcOCHMe(CH2)3CO2Ac, b22 123-6° in a tube at 240° the product is pentenyl acetate, b. 149-50°, this is hydrolyzed to a 1:1 mixt. (shown by KMnO4 oxidn.) of 3- and 4-penten-1-ol, b. 140-1°. Tetrahydrofurfuryl alc., AcCl, Zn dust, and C6H6 give a diacetate, b26 158-60°, either AcOCH2CH2(OAc)(CH2)2CH2Cl or AcOCH2CHCl(CH2)2CH2CO2Ac; XXXVa similarly gives ClCH2CH2CH2CO2Ac, b24 92-4°. A mixt. of 3 mol HCN and 1 mol X11a passed at 40 mL/h. over Al2O3 at 425° gives product contg. (mol. wt.) 13 X11a, 38 CH2=CHCH2CH2CN, b. 180-21°, 26 C8H5N, and 9 neutral and 12 basic high boiling fractions. XLI (650 g.), 83 g. PCl3, and 17 g. red P chlorinated at 120-60° and irradiated with a 500-w. light gives CH2ClCHClCH2CH2Cl, b7 74°, CH2ClCCl2CH2CH2Cl, b7 86-8°, CH2ClCCl2CHClCH2Cl, b7 102°, (CH2ClCCl2)2, b7 115°, and CHCl2CCl2CCl2CH2Cl, b7 125°. PhOH (188 g.) and 83 g. NaOH heated to 150° and 127 g. XLI added give, after 1 h., 120 g. PhO(CH2)4OPh, m. 97-8° (from alc.). XLI (380 g.), 700 mL. BuOH, and 200 g. KCN at 100-20° give 140 g. unchanged XLI, 42 g. NC(CH2)4CN (XLI1), and 146 g. CH2Cl(CH2)3CN (XLIIV), b16 100-3° Compsd. prepd. from XLIIV are (reagents, time, and temp. in parentheses): 6-valerolactone, b18 110-15° (hydrazide, CSH2NH2O2, m. 107°), and its polymer (H2O, 4 h., 180°); piperidine (NH3, Raney Co, and H, 6 h. at 70°, 100 atm.); piperidone together with N-4-cyanobutylpiperidone, b10 200-15° (NH3 at 70-120° in an autoclave, low yield); Et2N(CH2)4CN, b45 130-5° (Et2NH 6-10 h. at 100-20°); Et2N(CH2)5NH2, b27 103-5° (Et2NH and Raney Co under N 6-8 h. at 120°, then H at 100 atm.); BuNH(CH2)5NH2, b20 165-8° (BuNH2 under N 4 h. at 100-10°, then Raney Co and H at 80-100°, 200 atm.); PhNH(CH2)4CN, b17 155-65° (PhNH2 overnight at 110°), this with Raney Co and H at 100°, 200 atm. gives PhNH(CH2)5NH2, b17 133-5°; o-MeC6H4SO2NH(CH2)4CN, b10 296-8° (procedure not given); and cyclobutyl cyanide, b. 149°, b25 55° (by refluxing with NaNH2 in dry Et2O; this with 50% NaOH gives the acid, b. 190-1°). 8-Valerolactone (250 g.) and 100 g. NaCN heated to 230°, the melt cooled and dissolved in H2O affords the solution subsides, and the soln. decolorized with C, acidified, and extd. with Et2O gives 120 g. NC(CH2)4CO2H, b1 162-70°; the Et ester, b24 145-50°, gives with alc. . NH3, Raney Co, and 200 atm. H at 130° e-caprolactam, m. 65°, b15 140-2°. XLI11 (720 g.), 720 g. dry NaCN, 30 g. CaCO3, and 5 g. NaHCO3 heated to 160°, 800 g. XLI added so that the temp. remained at 180-200°, and the mixt. heated another 2 h. give 600 g. newly formed XLI11. NH2(CH2)5CN, b10 106° (Bz deriv., m. 96-7°), (200 g.) is prepd. from 440 g. XLI11, 130 g. NH3, and 30 g. Raney Ni under N at 130° and 120-40°; then Raney Co and H at 80-100°, 200 atm., the product is H2N(CH2)6NH2, b10 81.5°, m. 45-6° (N,N'-diformyl deriv., m. 112°; N,N'-di-alc deriv., m. 127°). XLI (126 g.) added at 80-5° to 150 g. anhyd. Na2S in 340 g. H2O and 100 mL. alc. and stirred 18 h. at the same temp. gives 44 g. tetrahydrothiophene, b. 119-22°. O[(CH2)4R]2 prepd. from O[(CH2)4Cl]2 are (R, const., reagents, and conditions given in parentheses): OH, b15 150-5° (8% Na2CO3 2 h. at 150°) (diacetate, b2 139-60°); HCO2, b0.8 120-30° (aq. alc.); NaOCH2 2 h. at 140°; SH, b0.6 113° (aq. Na2S, satd. with H2S, 10 h. at 145°); NH2, b1.4 105-8° (large excess NH3,

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 (212 g.), 230 g. CH2Br(CH2)2CHBrMe, and 45 g. AlCl3 at 90-100° give 130 g. of a mixt. (b13 90-130°) of (apparently) 1,5,7- and 4,5,7-trimethyltetrahydronaphthalene. C6H6 (312 g.), 20 g. AlCl3, and 254 g. XLI1 give 32 g. hexahydrotriphenylene. A polymeric oxo acid is prepd. from xylene, AlCl3, XLI, and phthalic anhydride. Naphthalene (1500 g.), 570 mL. X11a, 20 g. CoI2, and 3 mL. AcOH heated 6 h. at 280-90° under 100 atm. CO gave phenanthrene, m. 100° (picrate, m. 145°), and fractions, b1 95-123° (contg. Cl4H16, Cl4H14, and Cl4H12 isolated chromatog. on Al2O3, and not further characterized), b1 141-2° (probably dinaphthylbutyl ether, C28H30O), and b1 247-60° (naphthylbutanol, Cl4H16O). A mixt. of 720 g. 70% CH2=CHCH2OH and 870 g. 35% H2SO4 added to 100 mL. 0.5% OsO4 (pH adjusted with acid to 3-4), with simultaneous addn. of KOH to hold the pH at 3.5-3.75 gives glycerol in 90% yield. meso-Erythritol, prepd. similarly, m. 120° (from MeOH). Cl (100 g.) added to 140 g. 70% CH2=CHCH2MeOH in 860 mL. H2O at 10-20°, the mixt. stirred overnight, excess Cl swept out with air, 500 mL. 13% Na2CO3 added, and the mixt. evapd. gives 100 g. HOCH2CH(OH)2Me, b1 140-2°. Di-Erythritol (dibenzal deriv., m. 218-19°) is prepd. similarly from HOCH2CH(OH)CHClCH2OH, b1.5 145° (decompn.), this, with 30% H2SO4 gave 3,4-dihydroxy-X11a, b4 121-2°. XXXVa chlorinated in CCl4 at 10° gives 3,4-dichloro-X11a, b14 59-61°. HgO (135 g.) in 500 mL. H2O treated with 85 g. Cl with cooling, the mixt. filtered and distd. in vacuo, and 35 g. XXXVa added with cooling to the distillate (which contained HOCl) gives 36 g. 3-chloro-4-hydroxy-X11a, b14 102-3°, this (50 g.) added in vacuo to warm milk of lime and the vapors condensed and redistd. gives 24 g. 3,4-oxido-X11a (XLVII), b. 143°, b14 45°. XLVII, ROH, and Al2O3 at 100° gave monoesters of 3,4-dihydroxy-X11a (R and const. given): H, b13 163°; Me, b1 107°; Et, b12 112°; Bu, b15 134°; HO(CH2)4, b12 201°; Ph, b0.5 131°; PhCH2, b0.6 162°; 2,5-dimethyl-3-hydroxy-4-methoxy-X11a, b10 102°. XLVII (86 g.) cooled and 400 g. 25% NH3 added gives 3-hydroxy-4-amino-X11a, b11 142°, m. 78°. Amino derivative, prepd. from XLVII and R1R2NH at 200° are (R1 and R2 given): octadecyl, H, b0.5 205°; Ph, H, m. 105° (from ligroine); Ph, Me, m. 63-4°, b2.5 175°; pyrrolidine, b1 153°. XXV bis(tetrahydro-3-furyl) ether, b2 145-50°, and 71 g. Cl added simultaneously at 15-20° during 2 h. to 2300 mL. H2O give 284 g. crude chlorohydrin which, added to 74 g. powd. Ca(OH)2 in 500 mL. H2O, gives 150 g. 2,3-oxido-1,4-butylene bis(tetrahydro-3-furyl) ether, b1 162-70°. XXV (665 g.), treated at 5-10° with 247 g. HCl gives 300 g. 3-chloro-X11a, b17 70-5°. XLI1 (280 g.) and 9 g. powd. "Na dibutylphthalenesulfonate" in 10 l. H2O treated at room temp. with 205 g. Cl dild. with air gives an unidentified tetrachlorobutane, b20 93-5°, and 287 g. CH2ClCHClCH(OH)CH2Cl (XLI1a), 2 isomers, b1.2 74-6° and b1 103-4°. XLI1a (142 g.) and 60 g. powd. Ca(OH)2 in 70 mL. H2O shaken 15 min. gives 70 g. of a mixt., b3 68-71°, of 1,4-dichloro-2,3-oxidobutane and 1,2-dichloro-3,4-oxidobutane. H2O2 (110 g. 40%) added to 125 g. XLI1, 200 g. AcOH, and 0.04 g. OsO4 gives, after 20 h., 56 g. CH2ClCH(OH)CH2CH2Cl, m. 127°, this (80 g.) in 1 l. X11a refluxed 3 h. with 60 g. NaOH gives 30 g. 1,2,3,4-dioxidobutane, b23 50-1°. XXV (200 g., 93%) in 1 l. AcOH chlorinated 5 h. at 0-10°, gives 280 g. (CHClCH2OH)2, b1 134-6°, m. 70°. (CHBrCH2OH)2, prepd. similarly in CH2Cl2, m. 87° (from C6H6), b1.3-1.5 148-50°. HCl passed into 400 g. HOCH2CH(OH)CH2CH2OH and 8 g. AcOH at 80-100° until 300 g. has been absorbed gives 420 g. CH2ClCH(OH)CH2CH2Cl, b18 95-100°; 400 g. of this in 550 mL. H2O and 112 g. NaOH in 250 mL. H2O at 100° give 240 g.

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 alc., and alkali earth oxide a short time at 100-20°; cyano, b1 172-6° (aq.-alc. NaCN 5 h. at 130°) (hydrogenation gives O[(CH2)5NH2]2, b1 137-8°). [HO(CH2)4]2O (720 g.) or (CH2CH2OH)2, and 240 g. NaOH heated to 180°, 800 g. O[(CH2)4Cl]2 added to the mixt. stirred 0.5 h., distd. with H2O, neutralized, and the oil distd. to about 250°/0.5 mm. gives 700 g. residual dihydroxypolybutyl ether, HO no. 170; (CH2CH2OH)2 and O[(CH2)4Cl]2 give a similar product. Longer heating gives higher-mol.-wt. products. O[(CH2)4Cl]2 in anhyd. C6H6 treated with Na at 100-5° (cooling) gives H(O(CH2)8)OH 50 g. of this, added to 200 g. 65% HNO3 at 50-5° give HO2C(CH2)6CO2H, m. 138°. XLI1 (560 g.) treated during 6 h. with 390 g. Cl at -10° gives 664 g. CH2ClCHClCCl2Me, b. 203-6°, b18 92-3°, and 110 g. (CH2ClCHCl)2 (XLY), m. 73° (from ligroine), b50 130-40°, XLI1 (250 g.) and 10 g. Bz2O2 heated to 80°, 550 g. SO2Cl2 added, and the mixt. warmed 3 h. at 80-100° give 140 g. XLV, 170 g. CH2ClCCl2CH2CH2Cl, b8 86-8°, and 35 g. pentachlorobutane, b7 102°. (CH2CH2OR)2 prepd. by refluxing XLI1 and NaOH or KOH in ROH are (R given): Me, b.141°; Et, b12 61-3°; iso-Pr, b12 80-2°; CH2=CHCH2, b20 106°; Bu, b18 128°; Ph, b1 175° (decompn.); MeC6H4, b0.4 170-6°; cyclohexyl, b15 160°; PhCH2, b1 137-9°; tetrahydro-3-furyl, b2 148°; tetrahydrofurfuryl, b2 150°, b3 105-6°, and the bis-MeOCH2CH2 ether, b15 105-6°, and the bis-BuOCH2CH2 ether, b2 148-50°, are prepd. similarly. XXV (500 g.) treated with 160 g. powd. NaOH (cooling), 70 mL. H2O distd. in vacuo, 250 g. XLI1 added slowly with stirring at 110-20° and the temp. held 2 h. gives viscous, nondistillable, H2O-sol. tri(butenediol), HO no. 480; diacetate, Cl6H24O6, b1 78-82°. XLI1 (500 g.) added to 420 g. XXV and 350 g. NaOH refluxing in 900 g. X11a 2 h. the solvent distd., and the residue allowed to crystallize gives XXV cyclic diether, C8H12O2, m. 117°, b. 184°, hydrogenation over Pd at 120° gives the satd. cyclic diether, C8H16O2, b. 183-5°. (CH2CH2R)2 prepd. from XLI1 were (R and reagents, and conditions in parentheses given): morpholino, b0.9-1.1 141-2° (morpholine 1 h. at 100°); 1-pyrrolidinyl, b0.5 98-105° (pyrrolidine several hrs. at 90-100°); 2-benzothiazolylthio, m. 86° (from alc.) (2-mercaptobenzothiazole and NaOH in X11a); NaO3S, no const. (aq. Na2SO3 7 h. on H2O bath); and CN, b14 150-80°, m. 76° (from alc.) (alc. NaCN refluxed 5 h.). The latter compd. decomps. in H2O, and the residue is distd., even in the presence of methylene blue. XLI (500 g.) added with stirring at room temp. during 8 h. to 700 mL. C6H6 and 80 g. anhyd. AlCl3 and heated to 80° until HCl evolution ceases gives 10 g. tetrahydronaphthalene, b0.3 45°, 20 g. Ph(CH2)4Ph, b0.2 70-85°, 60 g. of a mixt. (b0.2 85-100°) of octahydroanthracene and phenanthrene, 25 g. octahydro-anthracene, m. 70°, b0.2 100-30° (this with MnO4 gives pyromellitic acid), and, from the residue, 68 g. dodecahydrotetraphenylmethane (XLI1V). XLI1 (460 g. from 950 g. XLI and 500 mL. C6H6 treated with 500 g. anhyd. AlCl3 during 48 h. below 20°), m. 239° (from PhCl), fluoresces blue-white in UV light. Similarly prepd. in low yield are: from PhMe, a mixt., b1 140-5° of 9-methyl-1,2,3,4,5,6,7,8-octahydroanthracene and 9-methyl-1,2,3,4,5,6,7,8-tetrahydronaphthalene; from m-C6H4Me2, dimethyltetrahydronaphthalene, b0.25 86-7° (dehydrogenation over ZnO gives dimethylnaphthalene; picrate, m. 118-19°), and Cl6H22, b0.3 156°, m. 132° (dehydrogenation gives anthracene and a hydrocarbon, m. 87°, from Cl6H8 a mixt. (b1 100-20°) of tetrahydroanthracene and phenanthrene and some (4-chlorobutyl)naphthalene, b1 140-50°; from Ph2 a resin. m-C6H4Me2

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 1-chloro-3,4-oxidobutane, b45 63°, 190 g. of which heated to 120-30° with 90 g. powd. NaOH gives 100 g. butadiene monoxide, b. 68°. XXV (88 g.) and 89 g. anthracene in 3 h. at 250° under N gives 9,10-endo-(2',3'-butane-1',4'-diol)anthracene (XLI11), m. 221° (from alc.); diacetate ester, m. 135° (from alc.-H2O). Similarly XXXVa gives, with anthracene, 9,10-endo-(3',4'-tetrahydrofuryl)anthracene, m. 180° (also obtained from XLI11 and 35% H2SO4 refluxed 7 h.), and, with 9,10-dichloroanthracene, 9,10-dichloro-9,10-endo-(3',4'-tetrahydrofuryl)anthracene, m. 165°. [MeCH(OH)CH]2 analogously gives 9,10-endo-(3',4'-hexane-2',5'-diol)anthracene, m. 213°. X11a (10 g./h./l. catalyst) and NH3 (molar ratio 1:20, resp.) passed over Al2O3 at 300-20° gives 80-5% pyrrolidine (XLI1X), b. 87-8° (N-alc deriv., b. 112-14°, p-O2NC6H4CO deriv., m. 92-3°), 1-(3-butenyl)-XLI1X, b. 152-4°, b14 64-5° (picrate, m. 105° (from alc.)), 1-(4-aminobutyl)-XLI1X, b. 205° (picrate, m. 126° di-Bz deriv., b1 180°), 1,4-di-(1-pyrrolidinyl)butane, b1 100° (picrate, m. 158°; dihydrate, m. 70°); N(CH2CH2CH2CH2)3, b1 120°. HO(CH2)4OH similarly gives 85% XLI1X and 6-8% high-boiling compds. contg. HN(CH2CH2CH2)2, b. 155-60° (picrate, m. 248°). XLI1X or HO(CH2)4OH and RNH2 over Al2O3 or ThO2-Al2O3 at 300-20° give the following 1-derivs. of XLI1X (substituent given): Me, b. 79-81°; Et, b. 105-7°; Pr, b. 130-5°, b15 32-5°; Bu, b. 154-6°, b15 48-50°; n-C10H21, b1 114-16°; CH2CH2NH2, b10 55-7°; 1,2-di-(1-pyrrolidinyl)ethane (formed with the preceding compd.), b9 98-100° (hydrate, m. 50°); Ph (l), b1 102-4°, m. 11° (L couples with diazo compds., with HNO2 L gives a green p-ON deriv., m. 128°); α-naphthyl, b0.4 137°; cyclohexyl, b. 213-15°; 2-pyridyl, b11 120-1°; 3-ethyl-6-methyl-2-pyridyl, b10 120-30°; 4-quinolyl, b0.8 158-60°. Analogously 222 g. HOCH2CH(OH)CH2CH2OH and 205 g. PhNH2 give 76 g. 1-phenylpyrrolidine. [MeCH(OH)CH2]2 gives 1-phenyl-2,5-dimethyl-XLI1X, b0.6 80-60°; tetrahydrofuran with NH3 gives piperidine and 1-(pentenyl)piperidine, b13 83-8°, PhNH2 gives 1-phenylpiperidine, b15 135-6°. L (27 g.) added at 10° to 50 g. PhNMeCHO and 69 g. POC13, the mixt. warmed several hrs. to 40-5°, and decompd. with ice gives 4-(1-pyrrolidinyl)benzaldehyde, m. 81° (from Et2O-petr. ether); semicarbazone, m. above 300°. Similarly L and COCl2 in CHCl3 at 5° give 4-(1-pyrrolidinyl)benzoyl chloride, subliming at 100-50°/0.1 mm. hydrolysis gives the acid, m. 294-5° (from alc.). HOCH2CH(OH)CH2CH2OH (160 g.), 115 g. PhNH2, and 160 g. PhNH2.HCl heated 12 h. at 190° gives 50 g. 3-hydroxy-L, b1 138-44°; 1-m-tolyl-2-hydroxy-XLI1X prepd. similarly from m-MeC6H4NH2, b0.7-0.5 131-40°; 2-methyl-L, 100 g. from 172 g. XXV, 230 g. PhNH2, and 30 g. fullers earth 12 h. at 270°, b13 125-35°. XLI1X (93 g., 90%), 66 g. m-MeC6H4OH, 60 mL. concd. HCl, and 200 mL. H2O 12 h. at 160° give 50 g. 1-(m-hydroxy)-L, m. 128°; 1-β-naphthyl-XLI1X, m. 92°, 250 g. from 216 g. β-naphthol, 200 g. XLI1X, and 20 g. fullers earth 6 h. at 200°; 2,6-di-(1-pyrrolidinyl)naphthalene, m. 240°, was prepd. similarly. In 90 g. yield from 70 g. 2,6-dihydronaphthalene and 80 g. XLI1X. Ethylene oxide (246 g.) passed at below 50° into 355 g. XLI1X and warmed 1 h. at 50° gives 213 g. 1-(2-hydroxyethyl)-XLI1X, b1 57-62°, 102 g. 1-(2-(2-hydroxyethoxy)ethyl)-XLI1X, b1 92-4°, and 29 g. 2-(2-hydroxyethoxy)ethyl β-1-pyrrolidinylethyl ether, b1 120-4°. VIII (100 g. 30%), and 142 g. XLI1X in 1 h. at 50° give 130 g. di(1-pyrrolidinyl) methane, b10 82-5° (1-Me-XLI1X gives bis(2-methyl-1-pyrrolidinyl) methane, b1 57-8°). The following

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 1-substituted XLIX are prep'd. (substituent given): CH₂CH₂CO₂Me, b. 5
 66-68°; CH₂CH₂CH₂CO₂Me, b. 105-6° [this with 100 atm. H at
 100° over Co gives amine, C₇H₁₃N₂, b. 105-6°
 (monopicate, m. 145°; mono-p-anilobenzoyl deriv., m.
 138°); CH₂CO₂Me, b. 112-14°, and a comp'd. contg. 2 mol XLIX and
 1 mol CO (from XLIX and CO), COMH₂, m. 218° (from H₂O). CS₂ (144
 g.) and 213 g. XLIX treated with Me₂SO₄ give 100% N-
 pyrrolidinyldithiocarbamic acid Me ester, b. 155-8°, m. 90°.
 p-Toluenesulfonylurethane (197 g.), and 71 g. XLIX, react on mixing to give
 N-(1-pyrrolidinyldithiocarbonyl)-p-toluenesulfonamide, C₁₂H₁₆N₂O₃S, m. 214°
 (from alc.). XLIX (284 g.), and 142 g. (CH₂ClCH₂)₂O heated 12
 h. at 160° give 170 g. bis(2-(1-pyrrolidinyldithio)ethyl) ether, b. 22
 166-72°. The following comp'ds. are prep'd. by conventional methods
 from XLIX and halo (usually Cl) comp'ds. (R = 1-pyrrolidinyldithio): (CH₂CH₂CH₂)₂
 b. 146-52°; (HOCH₂CH₂CH₂)₂, b. 190-200°, m. 61°
 p-O₂N₂C₆H₄R (Li), m. 166° (hydrogenation over Ni-Cr₂O₃ at
 70-80°, 200 atm. p-H₂N analog (Li), b. 140°, m.
 35°); p-O₂N₂C₆H₄CH₂R, hydrogenated to the p-NH₂ analog, b.
 129°, m. 51° (from ligroine); 1-(3-quinolyl)pyrrolidine, m.
 96-8° (from ligroine); m-O₂N₂C₆H₄SO₂R; p-AcNH₂C₆H₄SO₂R, m.
 177° (from H₂O); p-H₂N₂C₆H₄SO₂R, m. 168° (from H₂O) (by
 hydrolysis of the preceding comp'd. with 5N H₂SO₄); 3,4-dichloro-3,4'-SO₂R, m.
 112° (from ligroine); 3,5-dichloro-3,5'-SO₂R, m. 146° (from
 70% alc.); 2,4-dichloro-2,4'-SO₂R, m. 153° (from
 ligroine); 3,4-dichloro-3,4'-SO₂R, m. 126°. LiI sulfate (80 g.) in 250 g.
 conc'd. H₂SO₄ and 115 mL. H₂O treated at 110° with 72 g. Li, and 95
 g. glycerin, H₂O dist'd. to raise the temp. to 140° and held there
 6-7 h. gives 1-(6-quinolyl)-XLIX, b. 145-70°, m. 117° (from
 C₆H₆-ligroine). The sulfonylides (20 g.) from HO₃SCl and 4,2-ClMe₂C₆H₃OH
 refluxed 2 h. with 40 g. XLIX give 2,5,4-ClMe₂(HO)₂C₆H₂SO₂R
 (R=1-pyrrolidinyldithio, m. 126° (from ligroine). 2-
 Mercaptobenzothiazole (334 g.) in 1500 mL. of H₂O treated with 426 g.
 XLIX, then 5 L. 15% NaOCl gives 2-benzothiazolylsulfenylpyrrolidine,
 C₁₁H₁₂N₂S₂ (LiI), m. 178°. VIII (400 g., 30%) and 288 g.
 iso-PrCHO added with stirring and cooling to 284 g. XLIX and stirred
 several hrs. at 40-50° give 2,2-dimethyl-3-(1-pyrrolidinyldithio)propanaldehyde, b. 145-85°, this with NH₃ and 150 atm. H
 at 150° over Ni gives 2,2-dimethyl-3-(1-pyrrolidinyldithio)propylamine, b. 55-7°. XLIX (200 g.) and 200 atm. H 12
 h. at 200° over 40 g. Raney Co give 27 g. 1-Bu-XLIX, b.
 150-4° (picrate, m. 125-7°), and a small amt. of (CH₂CH₂CH₂)₂
 (R = 1-pyrrolidinyldithio), b. 138°, similar reaction in the presence of
 200 g. NH₃ gives 50 g. 2-Bu-XLIX, b. 154-6° (picrate, m.
 122-3°); at 300° over Raney Ni H₂N(CH₂)₄NH₂ and
 1-H₂N(CH₂)₄-XLIX, b. 89-119° (picrate, m. 125°), and
 1,4-di(1-pyrrolidinyldithio)butane, b. 121-6° (picrate, m. 157°),
 are formed. IX (190 g. 50% aq. soln.), sat'd. with NH₃ and passed during 3
 h. over 250 mL. 5% ThO₂ on Al₂O₃ over which 200 L/h. NH₃ is circulated
 gives 61 g. unchanged IX and 30 g. pyrrole (LiV), b. 130°. A mixt.
 contg. 2/3 1-Me-LiV, b. 112-13°, and 1/3 2-Me-LiV, b.
 147-8°, is prep'd. in 62% yield from 1-Me-XLIX passed at 500°
 with N over a ZnO-CuO-Al₂O₃ catalyst. 2-Me-LiV is also prep'd.
 in 84% yield from 1-Me-LiV over clay chips at red heat; L dehydrogenated
 similarly gives 80% 1-Ph-LiV, m. 56-8°, rearranged as above to
 2-Ph-LiV, m. 122-3°. XXXVa (180 g.), (or an equiv. amt. of XXV or
 HOCH₂CH(OH)CH₂CH₂OH) and 228 g. PhNH₂ over ThO₂-Al₂O₃ at 300° give
 1-phenyl-2,5-dihydropyrrole, b. 85-93°, m. 88° (from MeOH);
 this, 50 g., in 200 mL. C₆H₆ and 10 g. PhMeCHO and 110 g. POCl₃ in
 100 mL. C₆H₆, held 2-3 h. at 0-10° and 12 h. at room temp., gives

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 AB In the hope that Raney Cu as a hydrogenation catalyst
 might help to resolve problems of selective reduction, it was prepared with
 the same care and under similarly varied conditions as Raney Ni. The
 alloy containing 50% Al, 45% Cu, and 5% Zn was powdered
 and separated into 170-, 270-, and 325-mesh particles. Catalyst A
 was prepared according to Fauconneau (C.A. 31, 3217.1). Adding in small
 portions during 20 min. 30 g. of the alloy of a given mesh to a stirred
 and refluxed (at a constant temperature) solution of 60 g. pure NaOH in 140
 cc. H₂O.
 Keeping the mixture at the same temperature 50 min., cooling, decanting the
 solution,
 and washing the catalyst with 12-15 L. distilled H₂O, twice with
 100 cc. alc., and 3 times with 100 cc. Me₂CO gives
 catalyst B, kept under Me₂CO. The reductions were carried out in
 a Parr bomb capable of withstanding 400 atm./sq. cm. at temps. up to
 400° with com. electrolytic H₂ from a cylinder under 150 atm. (the
 amount of compound to be reduced, its a.p. or b.p., weight of catalyst
 and in parentheses the temperature at which it was prepared and its mesh
 value),
 H absorbed (from difference between initial and final pressure), time and
 temperature of heating, product, its a.p. or b.p. and % yield are: 0.33 mole
 cyclohexene, b. 82.5°, 4 g. A (0° and 170°), 0.34 mole H, 1 hr.,
 170-200°, cyclohexane, b. 80°, 100°, 0.33 mole anethole, b. 15
 109°, 4 g. A (0° and 170°), 0.33 mole H, 40 min.,
 150-80°, p-MeOC₆H₄Pr, b. 120-90°, 100°, 0.15 mole PhCH:CHCH₂OH,
 m. 33°, 2 g. A (0° and 170°), 0.18 mole H, 1 hr.,
 170-210°, Ph(CH₂)₃OH, b. 140°, 100°, 0.05 mole anthracene
 (I) (in 150 cc. PhMe), -, 2 g. B (90° and 325°), -, 45 min.,
 250°, 9,10-dihydroanthracene (II), m. 170°, 100°, 0.2 mole I
 (in 100 cc. PhMe), -, 4 g. B (60° and 270°), 0.2 mole H, 2 hrs.,
 300°, 11, -, 100°, 0.15 mole phenanthrene, m. 99°, 5 g. B
 (90° and 325°), 0 mole H, 1 hr., 300°, -, -, 0.2 mole
 C₁₀H₈, m. 80°, 2 g. A (0° and 170°), 0 mole H, 2 hrs.,
 300°, -, -, 0.45 mole AcEt, b. 79°, 2 g. A (0° and
 170°) (activated by 0.15 cc. 40% NaOH), 0.48 mole H, 40 min.,
 200-30°, 2-butanol, -, 100°, 0.32 mole iso-PrAc, 3 g. A
 (0° and 170°) (activated by 0.15 cc. 40% NaOH), 0.3 mole H, 1 hr.,
 150-70°, iso-PrCH(OH)Me, -, 100°, 0.24 mole (iso-Bu)₂CO, m.
 168°, 3 g. A (0° and 170°) (activated by 0.3 cc. 40% NaOH),
 0.22 mole H, 2 hrs., 180-200°, (iso-Bu)₂CHOH, b. 173°, 95°
 0.5 mole PrCHO, b. 75°, 4 g. A (0° and 170°) (activated by
 0.2 cc. 40% NaOH), 0.51 mole H, 40 min., 200-30°, BuOH, -, 100°, 0.5
 mole MeCH:CHCHO, b. 101°, 3 g. B (60° and 170°) (activated by
 0.4 cc. 40% NaOH), 1.04 mole H, 75 min., 180-200°, BuOH, b.
 116°, 100°, 0.5 mole Me₂C:CHAc, b. 130°, 3 g. A (0°
 and 170°) (activated by 0.3 cc. 40% NaOH), 0.98 mole H, 1 hr.,
 170-200°, iso-PrCH₂CH(OH)Me, b. 131°, 95°, 0.57 mole
 cyclohexanone, b. 155°, 3 g. A (0° and 170°) (activated by
 0.2 cc. 40% NaOH), 0.56 mole H, 40 min., 160-80°, cyclohexanol, b.
 159°, 100°, 0.33 mole isophorone, b. 93°, 3 g. B (90°
 and 325°), 0.67 mole H, 75 min., 150-70°, 3,3,5-
 trimethylcyclohexanol, m. 54°, 100°, 0.1 mole PhCH:CHAc, m.
 39°, 2 g. A (0° and 170°), 0.19 mole H, 45 min.,
 180-210°, Ph(CH₂)₂CH(OH)Me, b. 127°, 90°, 0.042 mole
 (PhCH:CH)₂CO, m. 112°, 2 g. A (0° and 170°) (activated by 0.2
 cc. 40% NaOH), 0.11 mole H, 1 hr., 170-200°, [Ph(CH₂)₂CH(OH)Me]₂,
 44°, 90°, 0.23 mole Et₂H, -, 2 g. A (0° and 170°), 0.3 or 0.47
 mole H, 1 hr., 150-75° or 200-30°, 90% PhCH₂CH₂OH and 5% PhMe
 or PhMe, -, -, 0.05 or 0.2 mole PhAc, b. 92°, 3 g. A (0°

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 4-(2,5-dihydro-1-pyrrolyl)benzaldehyde, m. 90-1° (from
 cyclohexane); semicarbazone, m. 270° (from alc.). XIIa
 (100 g., 94%) added to 662 g. 65% HNO₃ and 4 g. NaNO₂ at 25° gives
 139 g. (CH₂CO₂H)₂, its anhydride (LiVa) is prep'd. by passing this over
 Al₂O₃ at 275° and 130-60 mm. HO₂CCH₂CH₂CO₂H is prep'd.
 similarly from 3-Me-XIIa; the anhydride, m. 30-5°, b. 105°.
 XXXVa (100 g.), passed at the rate of 6 g. and 240 L. air/h. over 100 mL.
 catalyst (prep'd. by heating 110 g. (NH₄)₂MoO₄, 32 g. vanadic acid,
 and 40 g. TiO₂ 1 h. with 1 L. 4% (CO₂H)₂, adding 30 g. NH₄Cl, then 500 g.
 pumice, evap'g., and heating 2 h. at 300° gives 120-30 g. maleic
 acid (LV) and anhydride; 100 g. XIIa similarly gives 80-90 g. LV and
 anhydride. Cl (70-80 g.) passed into 100 g. LiVa at 160-80° gives
 60 g. LV anhydride and 40 g. unchanged LiVa. Monochloro-LV anhydride is
 prep'd. from maleic anhydride (or LiVa), Cl, and FeCl₃ at 160-80°;
 100 g. LiVa, some FeCl₃, and 240 g. Cl at 160-80° give 150 g.
 dichloromaleic anhydride. LiVa (600 g. 80%), treated at 190-200°
 with 3 g. NaOEt gives 260 g. γ-oxopimelic acid dilactone, b.
 170°, 300 g. of this refluxed 60 h. with 600 mL. EtOH and 30 g.
 conc'd. H₂SO₄ give 305 g. diester of γ-oxo-pimelic acid, b.
 165°. Hydrogenation of the dilactone in alk. soln. at 160°,
 200 atm. gives 90% γ-hydroxypimelic acid; 500 g. of the dilactone,
 and 350 g. NaHCO₃ in 1500 mL. H₂O hydrogenated over 35 g. Raney Ni and 35
 g. Cu chromite at 280°, 200 atm. give 220 g. pimelic
 acid.
 ACCESSION NUMBER: 1956:89209 CAPLUS
 DOCUMENT NUMBER: 50:89209
 ORIGINAL REFERENCE NO.: 50:16777d-i,16778a-i,16779a-i,16780a-i,16781a-i,16782a-
 i,16783a-b
 TITLE: Ethynylation. V. Reactions of hydrated ethynylation
 products. Dehydration of γ-alkanedioles
 AUTHOR(S): Reppe, Walter; et al.
 SOURCE: Ann. (1955), 556, 60-158
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 50:89209

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 and 170° (activated by 0.2 cc. 40% NaOH) or 3 g. B (50° and 325°),
 0.6 or 0.39 mole H, 1 hr., 150-75° or 200-40°, PhEt, -, -, 10, 85, and 100; 0.1 or 0.5 mole, Ph₂CO, m.
 48°, 3 g. B (50° and 170° or 60° and 270°), 0.1 or 1.2
 mole H, 1 hr., 150° or 230-50°, Ph₂CHOH or Ph₂CH₂, -, -, 95
 and 100; 0.1 mole benzoin, -, 3 g. A (0° and 170°) (activated by 0.2
 cc. 40% NaOH), 0.11 or 0.29 mole H, 1 hr. or 20 min., 150° or
 250°, [PhCH(OH)]₂ or [PhCH₂]₂, -, -, 95 and 100; 0.2 mole RCH:CHBz
 (R = 2-furyl) (in 100 cc. MeOH), -, 2 or 3 g. B (50° and 325°)
 (activated by 0.15 cc. 40% NaOH), 0.5 or 0.6 mole H, 80 min.,
 160-80° or 200-30°, R(CH₂)₂CH(OH)Ph, b. 100° or
 R(CH₂)₃Ph, b. 100°, 95 or 100; 0.58 mole 2-furaldehyde, b. 5
 54°, 3 g. A (0° and 270°) or B (50° and 325°),
 (activated by 0.2 cc. 40% NaOH), 0.62 or 1.01 moles H, 50 min. or 2 hrs.,
 150-65° or 200-40°, furfuryl alc. (III), b. 15
 68°, or 111 and 2-methylfuran, 95, or 20 and 70; 0.3 mole PhCN, -,
 2 g. A (0° and 170°), 0.58 mole H, 1 hr., 180°, PhCH₂CH₂, -, -,
 and [PhCH₂]₂ 2NH, -, 40 and 45; 0.1 mole coumarin (in 50 cc. MeOH), 1 g. A
 (0° and 170°) or 2 g. B (60° and 170°), 0.07 or 0.3 mole H, 45
 min. or 2 hrs., 140-60° or 210-40°, hydrocoumarin, b. 20
 165°, or o-HOC₆H₄CH:CHCH₂OH, benzoate, m. 98°, 100 or 90;
 0.1 mole 2-naphthol, -, 2 g. B (50° and 170°) (activated by 0.2 cc.
 NaOH), 0.18 mole H, 2 hrs., 260-80°, tetrahydro-2-naphthol, b.
 117°, 80; 0.5 mole 1-naphthol, 3 g. A (0° and 170°)
 (activated by 0.2 cc. 40% NaOH), 0.24 mole H, 80 min., 270-90°,
 1,2,3,4-tetrahydronaphthalene, b. 204°, 40; 0.2 or 0.1 or 0.1 mole
 PhNO₂, 3 g. B (60° and 270°) or (90° and 325°) (activated by
 0.3 cc. NaOH) or (90° and 325°) (activated by 0.3 cc. Et₃N), 0.6
 or 0.27 or 0.27 mole H, 90 or 45 or 45 min., 170° or 140° or
 140°, PhNH₂ in all 3 cases, 100 in all cases; 0.072 mole
 p-O₂N₂C₆H₄NH₂ (in 50 cc. C₆H₆), 1 g. B (90° and 325°) (activated by
 0.4 cc. Et₃N), 0.24 mole H, 15 min., 240-60°, p-C₆H₄(NH₂)₂, 100;
 0.036 mole o-O₂N₂C₆H₄NH₂ (in 50 cc. C₆H₆), 1 g. B (70° and
 270°) (activated by 0.2 cc. Et₃N), -, 10 min., 220-50°.
 o-C₆H₄(NH₂)₂, -, 95; 0.072 mole m-O₂N₂C₆H₄NH₂ (in 25 cc. C₆H₆), 1 g. B
 (90° and 325°) (activated by 0.4 cc. Et₃N), 0.25 mole H, 15 min.,
 230-50°, m-C₆H₄(NH₂)₂, -, -, 0.15 mole p-O₂N₂C₆H₄NH₂ (in 50 cc.
 C₆H₆), 3 g. B (70° and 325°) (activated by 0.3 cc. 40% NaOH), 0.42
 mole H, -, -, p-H₂N₂C₆H₄OH, -, 100; 0.05 mole m-C₆H₄(NO₂)₂ (in 25 cc.
 C₆H₆), 1 g. B (90° and 325°) (activated by 0.2 cc. 40% NaOH or 0.2
 cc. Et₃N), 0.19 or 0.36 mole H, 30 or 15 min., 180-200° or
 250°, m-O₂N₂C₆H₄NH₂ or m-C₆H₄(NH₂)₂, -, -, -, 0.025 mole
 o-C₆H₄(NO₂)₂ (in 50 cc. PhMe), 1 g. B (60° and 325°) (activated by
 0.2 cc. Et₃N), 0.02 mole H, 35 or 25 min., 200° or 245°.
 o-O₂N₂C₆H₄NH₂ or o-C₆H₄(NH₂)₂, -, -, -, 0.025 mole p-C₆H₄(NO₂)₂ (in 50
 cc. PhMe), 1 g. B (60° and 325°) (activated by 0.2 cc. Et₃N), 0.02
 mole H, 30 or 40 min., 200° or 250°, p-O₂N₂C₆H₄NH₂ or
 p-C₆H₄(NH₂)₂, -, -, -, -. Thus hydrogenation in the presence of Raney
 Cu can be applied to the selective reduction of many types of
 comp'ds. contg. several reducible groups.
 ACCESSION NUMBER: 1956:88889 CAPLUS
 DOCUMENT NUMBER: 50:88889
 ORIGINAL REFERENCE NO.: 50:16651b-i,16652f-i,16653a-i
 TITLE: Catalytic hydrogenation in the presence of Raney
 copper
 AUTHOR(S): Jadot, J. Braine, R.
 CORPORATE SOURCE: Univ. Liege, Belg.
 SOURCE: Bull. soc. roy. sci. Liege (1956), 25, 62-78
 DOCUMENT TYPE: Journal

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AB A number of fluorophenothiazines which may be of interest as antioxidants in lubricating oils are prepared. Adding slowly with stirring 16 g. Br in 25 cc. AcOH to 12.9 g. 2,4-F2C6H3NH2 in 75 cc. AcOH at 25°, removing after 0.5 hr. any excess Br with Na2S2O3, then adding 11.2 g. NaOH in 100 cc. H2O, and cooling the mixture in an ice bath give 81% 2,4,6-F2BrC6H2NH2 (I), m. 41-2°. I has a high vapor pressure [N-Ac derivative (II), 90%, prepared with Ac2O, m. 156-7°]. 2,4-F2C6H3NHAc cannot be brominated in AcOH. Deamination of I with H3PO2 gives 74% 3,5-F2C6H3Br (III), b. 140°, d23 1.676, nD23 1.4989. Adding a Grignard reagent of 14.5 g. III and 1.9 g. Mg in 50 cc. Et2O to dry ice in Et2O gives 64% 3,5-F2C6H3CO2H, m. 121-2°, also obtained when 18 g. 3,5-(H2N)2C6H3CO2Et in 490 g. 45% HBF4 is treated at -10° with 15 g. NaNO2, the bis(diazonium fluoborate) (40 g., decomposing about 175°) is decomposed at 30 mm., and the Et ester, b46 103-5°, b760 200°, nD25 1.4670, d25 1.201, saponified with KOH. Stirring 23 g. 2,6-ClFC6H3CO2H in 100 cc. concentrated H2SO4 1 hr. at 60°, adding (1.5 hrs.) 10 g. NaBr in small portions at 65°, keeping the mixture overnight, making it basic with NH4OH, and steam distilling it give 70% 2,6-ClFC6H3NH2 (IV), b30 91°, nD23 1.5511, d23 1.316 (Ac derivative, prepared in 71% yield by refluxing 6 g. IV 1.5 hrs. in 25 cc. AcOH and 4.2 g. Ac2O, platelets, m. 134-5°). Adding 9.1 g. NaNO2 in small portions to 24 g. 2,3-O2N(H2N)C6H3CF3 in 300 cc. 50% H2SO4 at 0°, stirring the mixture 15 min., pouring it into 160 cc. 10% CuCl at 20°, keeping it 1 hr. at 20°, diluting it with 100 cc. H2O, and steam distilling it give 52% 3-Cl analog, b27 125-6°, nD24 1.4782, d24 1.531. 3,4-Cl(2O2N)C6H3CF3, prepared in the same way in 53% yield, b28 116°, nD24 1.4864, d24 1.527. Refluxing 17 hrs. 0.1 mole of the appropriate acetanilide, 0.2 mole PhBr, 0.1 mole anhydrous K2CO3, 20 cc. PhNO2, 6 g. catalyst mixture (consisting of equal parts by weight of CuI, XI, and Cu powder), and a crystal of iodine, steam distilling the mixture, extracting the distillation residue with Et2O, refluxing the Et2O residue 3.5 hrs. in 100 cc. 20% alc. KOH, pouring the solution into 800 cc. saturated NaCl solution, and extracting with Et2O give the fluorodiphenylamine of which the following are prepared: 2-F (V), 80%, yellow oil, b3 111.5°, d23 1.165, nD23 1.6171 (N-Bz derivative, m. 129-30°); 3-F (VI), 56%, yellow oil, b10 149-50°, d23 1.176, nD23 1.6203; 2,5-di-F (VII), 68%, b9 138°, m. 45-5.5°; 2,4-di-F (VIIa), 63%, b3 110-13°, m. 42-2.5°; 3,5-di-F (VIII), 51%, b4 121-4°, m. 45-5.5°; 2,3,5-tri-F, 63%, b2.5 105-6°, m. 31.5-2°; 2,4,5-tri-F, 71%, b2.5 104-5°, m. 39-2-40°; 3,3',5-tri-F (VIIIA), 49%, b3.5 121°, m. 27.5-8.5°; 3,4',5-tri-F (IX), 65%, b4 127-8°, m. 60-1°; 3,3',5,5'-tetra-F (X), 43%, m. 117-18°; 2,6-ClF (XI), 34%, b4 138-40°, m. 69-9.3°. Adding 4 g. NaOH in 30 cc. H2O to 12.5 g. o-H2NC6H4SH in 300 cc. absolute EtOH, then adding 0.1 mole of the appropriate halonitrobenzene in 100 cc. absolute EtOH, refluxing the mixture 0.5 hr. (3.5 hrs. in the preparation of XIII below), adding 100 cc. H2O to the boiling filtered solution, and cooling it slowly give the substituted o-H2NC6H4SC6H3RNO2-x,2 (XII) of which the following are prepared: R = 3-CF3 (XIII), 67%, m. 72-3°; 4-CF3 (XIV), 89%, yellow, m. 108-9°; 5-CF3, 80%, yellow, m. 110-11°; 4-F, 70%, red, m. 73-4°; 5-F, 54%, yellow, m. 115-16.5°. Refluxing XII with 10 times its weight of 90% HCO2H 9-10 hrs. gives the substituted N-formyl derivative, o-OHCNHC6H4SC6H3RNO2-x,2, of which the following are prepared: x-R = 3-CF3 (XV), 92%, m. 137-8°; 4-CF3 (XVI), 88%, yellow, m.

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AB Ph2NH (I) is obtained from PhNH2 (II) heated under pressure in the presence of a catalyst capable of liberating HCl. A mixture of II 400 g., CuCl2.2H2O (III) 12 g., powdered Fe (60 B.S.S. mesh) (IV) 12 g., and NH4Cl (V) 12 g. heated 3 h. to 350° in a Cu-lined, stirred autoclave vented to release the NH3 formed so that the pressure is maintained at 350-80 lb./sq. in., and the product cooled, filtered, and fractionated yields 53.4% I. This procedure carried out 3 times with reaction times of 2, 6, and 9 h. gives yields of I of 27.3, 63.7, and 68.0%, resp. Similarly, when other catalyst mixts. (12 g. each anhydrous CuCl, IV, and V; 12 g. each III, IV, and PhNH2.HCl (VI); 12 g. each, III, Zn dust, and V; and 12 g. III, 12 g. V, and 6 g. powdered Cu-bronze) are substituted, the yields of I are 48.0, 50.3, 38.3, and 47.5%, resp. After completion of the same process with III 8 g., IV 4 g., and VI 12 g., the catalyst filtered off, washed with ether, dried, mixed with VI 12 g., used again, and this procedure repeated until 4 runs are completed, gives yields of I of 54.7, 55.8, 56.3, and 54.8%, resp. When this procedure is carried out without adding VI after the 1st run, the yields of I are 51.9, 48.7, and 35.7%, resp.
ACCESSION NUMBER: 1956:57054 CAPLUS
DOCUMENT NUMBER: 50:57054
ORIGINAL REFERENCE NO.: 50:10769d-f
TITLE: Diphenylamines
INVENTOR(S): Weight, Donald; Wilde, Bertrand E.
PATENT ASSIGNEE(S): Monsanto Chemicals Ltd.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 738986		19551026	GB	

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132-3°, 5-CF3 (XVII), 83%, yellow, m. 95-6°, 4-F (XVIII), 74%, yellow, m. 128-9°; 5-F (XIX), 74%, yellow, m. 116-17°. Refluxing 6.3 g. XIV 1 hr. with 2.8 g. BzCl in 25 cc. CSH5N gives 89% 2-(4,2-CF3(2O2N)C6H3S)C6H4NHBz (XX), m. 127.5-8°. Heating 15 g. V, 5 g. S, and a few crystals of iodine 3 hrs. at 200-10° under reflux, boiling the tar obtained with 20% Na2S (to remove the excess S), extg. it with Et2O, treating the Et2O soln. with Norit and Zn dust, and distg. the residue of the filtered Et2O soln. give 2.2 g. unchanged V and 3 g. of a yellow solid, b2.5 140°, from which 9.3% 1-fluorophenothiazine (XXI), m. 81.5-2°, and 1.4% phenothiazine (XXII), m. 180-2°, are isolated. XXI gives a blood-red color with concd. HNO3. When 1.9 g. V, 0.6 g. S, and a crystal of iodine are heated 1.5 hrs. in a sealed tube at 310-40° a few mg. XXI and 0.175 g. XXII are obtained. When 2.2 g. XI, 0.6 g. S, and a crystal of iodine are heated 1.5 hrs. at 300°, no cryst. product can be isolated. Heating 5 g. VI, 1.7 g. S, and a few crystals of iodine 1 hr. at 180° ± 5° gives 52% 1-fluorophenothiazine (XXIII), light yellow powder, m. 199° (decomp.); it gives a blood-red color with HNO3. In an attempted synthesis of 1,4-difluorophenothiazine (XXIV), XXIII is obtained. Several other attempts to prep. XXIV from VII, from VIIa, or 1,3-difluorophenothiazine also failed. Heating a carefully purified VIII with S and a crystal of iodine 35 min. at 175° gives 43% 2,4-difluorophenothiazine, subliming 130°/2.5 mm., m. 129-30°. Attempts to prep. 1,4,7- and 1,4,8-trifluorophenothiazines by ring closure of the appropriate trifluorodiphenylamines failed. Ring closure of IX with S and iodine 1 hr. at 190° gives 20% 2,4,7-trifluorophenothiazine, m. 147-8° (decompn.); 6.1 g. VIIa, 1.75 g. S, and a crystal of iodine 2.5 hrs. at 170° give 44% 2,4,8-trifluorophenothiazine, m. 142-3°; 1.2 g. X, 0.31 g. S, and a crystal of iodine 1 min. at 230° and 20 min. at 210° (or 1.5 hrs. at 210-40°) give 13% (or 20%) 2,4,6,8-tetrafluorophenothiazine, needles, m. 193-3.5°. Refluxing 15 g. XVI in 150 cc. Me2CO 0.5 hr. with 44 cc. N NaOH, evap. the soln. to dryness, and extg. the residue with boiling CCl4 give 52% 3-trifluoromethylphenothiazine, m. 217-18°, also obtained in much lower yield from XX. In a similar way XVII gives 59% 2-trifluoromethylphenothiazine, m. 189-90°, and XVIII gives 43% 3-fluorophenothiazine, m. 178-9°. Attempts to prep. 4-trifluoromethylphenothiazine by treating 4.6 g. XV in 46 cc. Me2CO with NaOH, or 2-fluorophenothiazine by refluxing 4.4 g. XIX in Me2CO with NaOH were unsuccessful. The infrared absorption spectra of these compds. are detd. and an attempt is made to see if a reliable method of structure detn. of fluorophenothiazines can be made with these spectra. It is found that this kind of structural data. should be approached with discretion.

ACCESSION NUMBER: 1956:44616 CAPLUS
DOCUMENT NUMBER: 50:44616
ORIGINAL REFERENCE NO.: 50:8653b-1,8654a-f
TITLE: The preparation of some fluoro- and trifluoromethylphenothiazines, and some observations regarding determination of their structure by infrared spectroscopy
AUTHOR(S): Roe, Arthur; Little, Wm. F.
CORPORATE SOURCE: Univ. of North Carolina, Chapel Hill
SOURCE: Journal of Organic Chemistry (1955), 20, 1577-90
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 50:44616

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 4,2,3,5-ClMe3C6H2OH (VIII), m. 103-4° [from light petroleum (b. 60-80°)]. Bromination of VIII gave 6-bromo-VIII, m. 144.5-5.0° (from dil. AcOH). 2,3,5-Me3C6H2OH (40 g.) and Zn(CN)2 (80 g.) in 400 cc. dry ether were ice cooled while dry HCl gas was passed in 1.5 h., 40 g. powd. AlCl3 was added in portions with continued stirring and HCl passed another 0.5 h. with cooling and 1 h. without cooling. After hydrolysis and steam distn. to sep. unreacted phenol and any O-aldehyde, the residue was extd. with hot dil. aq. EtOH, giving 65% 2,3,6,4-Me3(HO)C6H2OH, m. 146.5-7.5° (from dil. EtOH). NaCH2O2 (5 g.) was condensed with 8.9 g. (EtO2CCH2)2CO in 30 cc. abs. EtOH contg. Na (1 g.) as ethoxide. The resulting ester was not sepd. but hydrolyzed with methanolic KOH, and the acid pptd. with HCl. From the very crude material (3.6 g.), a small amt. of cryst. material was obtained from AcOH, which crystd. from EtOH gave 3,4,5,2,6-Me3(HO2C)2C6OH, m. 200-1° (decompn.).

ACCESSION NUMBER: 1956:24205 CAPLUS
 DOCUMENT NUMBER: 50:24205
 ORIGINAL REFERENCE NO.: 50:4965f-1, 4966a-g
 TITLE: Preparation of some methyl-substituted phenols and phenol alcohols
 AUTHOR(S): Fitzgerald, J. S.
 CORPORATE SOURCE: Div. Ind. Chem., C. S. I. R. O., Melbourne
 SOURCE: Journal of Applied Chemistry (1955), 5, 289-96
 CODEN: JACHAU; ISSN: 0021-8871
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 50:24205

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 AB Some methods for the preparation of 2,3,4,5-Me4C6H2OH have been investigated, the most useful being the Gattermann reaction with 2,3,5-Me3C6H2OH followed by Clemmensen reduction. Hydrogenolysis of phenol alc. over copper chromite catalyst has been shown to give methyl-substituted phenols without rearrangement; nuclear chlorine does not interfere and is not removed during hydrogenolysis. The general procedure for the preparation of these compds. is given below. In preparation of the phenol alc. about 50 g. (1 M proportion) of the phenol was dissolved by warming in 75 cc. water containing 20 g. (1.5 M proportions) NaOH, 50-75 cc. 37% HCHO (2-3 M proportions) added after cooling to room temperature or until crystallization began, the mixture cooled with ice 0.5 h., kept overnight at room temperature, the Na salt filtered off and dissolved in 400-800 cc. water, then cooled with ice and acidified with a slight excess of dilute AcOH. In most cases the phenol alc. separated as a crystalline solid, but derivs. of 2,3,5-ClMe2C6H2OH separated initially as oils. The phenol alc. (5 g.) in 25 cc. dioxane was shaken with 2 g. copper chromite in a Parr bomb under H at 3000 lb./sq. in., the temperature being raised to 150-60° in about 10-15 min., then taken up and held at 170-80° 2 h. or up to 200° 0.5 h. The mixture was cooled with continued rocking 1 h. in the furnace, the catalyst residue removed by centrifuging and washed with dioxane, the bulk of the solvent distilled off and the methylated phenol isolated by steam distillation. The chloromethylphenol (5 g.) in 100 cc. 5% NaOH was heated to 80° and 5 g. of Raney Ni-Al alloy were added in small portions with continuous stirring over a 1-h. period, the clear solution added to 50 cc. 5N HCl after centrifuging, and the phenol steam distilled out. To find whether the presence of nuclear Cl interfered with the hydrogenolysis, p-ClC6H4OH dialc. was prepared and reduced. The resinous material left after steam distillation was 4,2,6-ClMe2C6H2OH, 77% yield, m. 83° [from light petroleum (b. 50-60°)]; phenylurethane, m. 142-4°. On hydrogenolysis of the dialc. of 4,3,5-ClMe2C6H2OH, 40-75% yields of 4,2,3,5,6-ClMe4C6OH, m. 114-15.5° [from light petroleum (b. 60-80°)], were obtained; phenylurethane, m. 231-5° (from EtOH); monoacetate, m. 79-80° (from aqueous AcOH). Using both the method given, but with 2 mol of NaOH per mol of xylenol, and the ethoxide method of Holmes (Finn, et al., C.A. 45, 1537i), 6,3,5,2-ClMe2(HOCH2)2C6H2OH (I), m. 102-4° [from light petroleum (b. 60-80°)], was prepared from 2,3,5-ClMe2C6H2OH; diacetate, m. 91° (from MeOH, then AcOH). Hydrogenolysis of I gave 60-80% 6,2,3,5-ClMe3C6H2OH (II), m. 56-8° (from aqueous MeOH). Bromination of II in AcOH gave 4-bromo-II, m. 157-8°; phenylurethane, m. 199° [from EtOH, then light petroleum (b. 80-100°)]. From II and HCHO is prepared [5,2,3,6,4-ClMe3(HO)C6]2CH2, m. 204-6°; diacetate, 205-6°. The more soluble component from the acetylation of the crude I was 6,2,3,5,4-ClMe3(AcO)C6OAc (III), m. 109° (from aqueous MeOH). Hydrogenolysis of III yielded the corresponding phenol alc. (IV), m. 108-10° (from MeOH); phenylurethane, m. 145-6° (from light petroleum (b. over 120°)), then dilute EtOH. Bromination of IV in AcOH gave 6,2,3,4,5-BrMe4C6OH, m. 114-15°. A methanolic solution of paraformaldehyde and piperidine was condensed with 3,4,5-Me3C6H2OH to give 56% 3,4,5-trimethyl-2-piperidinomethylphenol, m. 103-5° (from MeOH). Piperidine and HCHO with 4,3,5-ClMe2C6H2OH (V) gave 80% 4-chloro-3,5-dimethyl-2-piperidinomethylphenol (VI), m. 142-3° (from EtOH). Morpholine and HCHO with V gave 65% 4-chloro-3,5-dimethyl-2-morpholinomethylphenol (VII), m. 125-6°. Hydrogenation of either VI or VII with copper chromite gave

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 AB Some derivs. of 5,6,7,8-tetrahydro-1'-phenyltriazolo(4',5':3,4)carbazole (I) have been prepared from the appropriate 5-amino-1-arylbenzotriazoles (II). II were synthesized by condensation of the appropriate ArNH2 with 2,4-(O2N)2C6H3Cl, reduction with Na2S in hot EtOH to 2,4-H2N(O2N)C6H3NHAr (III), conversion to the 5-nitro-1-arylbenzotriazole (IV), and reduction with Zn dust and aqueous alc. CaCl2. The following new compds. are reported (aryl and m.p. given): p-Cl-C6H4: II, 170-1° (from aqueous EtOH). o-MeOC6H4: III, 146-7° (from CCl4); IV, 152-3° (from EtOH); II, 149° (from aqueous EtOH). m-MeOC6H4: III, 151-2° (from EtOH); IV, 201° (from EtOH); II, 108-9° (from aqueous EtOH). o-MeC6H4: II, 93° (from EtOH) [5-AcNH analog, m. 204° (from HOAc)]. Heating 1 g. II (Ar = Ph) (IVa) and 0.6 g. 2-hydroxycyclohexanone gave a melt at 126°, which solidified at 132° and, when stirred with hot EtOH and recrystd. from cyclohexanone, yielded 55% 5-(2-oxocyclohexylamino)-1-phenylbenzotriazole (V), m. 169°, infrared bands in Nujol at 2.98 and 5.85 μ 1-aryl analogs of V prepared were (aryl, maximum temperature reached, % yield, and m.p. given): p-ClC6H4, 150°, 53, 200-1° (from cyclohexanone and from EtOH); o-MeOC6H4, 140°, 55, 129-30° (from EtOH); m-MeOC6H4, 145°, 46, 178° (from EtOH); p-MeOC6H4, 145°, 45, 178-9° (from EtOH); o-MeC6H4, 140°, 51, 106-7° (from EtOH). V (0.8 g.) heated with a few drops of concentrated HCl melted at 145-50° and resolidified, giving on recrystn. from EtOH 0.2 g. I, m. 221-2°. Analogs of I, prepared similarly but with small amts. of the dry amine-HCl as the catalyst, were (I'-aryl, reaction temperature, % yield, and m.p. given): p-ClC6H4, 180°, 70, 251-2°; o-MeOC6H4, 150°, 16, 193°; m-MeOC6H4, 180°, 30, 203-4°; p-MeOC6H4, 170°, 8, 245-6° [with HCl as the catalyst, only the HCl salt of II (Ar = p-MeOC6H4) was isolated]; o-MeC6H4, 160°, 53, 226-7°. No pure products were isolated on heating I or its analogs at various temps. and conditions. IVa (5 g.), 5 cc. AcCH2CO2Et, and a few drops of concentrated HCl left 12 hrs. at room temperature gave 5.5 g. Et 3-(1-phenylbenzotriazol-5-ylamino)crotonate (VI), m. 127-8° (from EtOH). VI (6 g.) heated 5 min. at 265°, the powdered product extracted with hot C6H6, and the residue crystallized from PhNO2 gave 2.7 g. 2-methyl-1'-phenyltriazolo(4',5':5,6)quinolin-4-one (VII), m. 325-35° (decomposition). VII (1.2 g.), 10 cc. POCl3, and 2 g. PCl5 refluxed 1.5 hrs., cooled, poured into ice and HCl, filtered after 1 hr., and made alkaline with NH3 gave 0.8 g. 4-chloro-2-methyl-1'-phenyltriazolo(4',5':5,6)quinoline, m. 183-4° (from aqueous EtOH). Similarly, from 1-(p-aminophenyl)benzotriazole were prepared 66% Et 3-(p-benzotriazol-1-ylamino)crotonate, purified by boiling in C6H6 with C, evaporating, and crystallizing from EtOH, m. 130°, and 45% 2-methyl-6-(benzotriazol-1-yl)quinolin-4-one, m. 320-40° (decomposition). IVa (1.1 g.) and 0.7 g. AcCH2CO2Et heated 2 min. at 180° and crystallized twice from EtOH gave 1.2 g. 5-acetoacetamido-1-phenylbenzotriazole (VIII), m. 210° (decomposition). VIII (1.3 g.) heated 20 min. at 100° with 25 cc. concentrated H2SO4 and poured into H2O gave 1 g. sulfate of IVa but no 2-quinolone. VIII (2.5 g.) refluxed 4 hrs. with 12 cc. POCl3, diluted with 150 cc. CHCl3, poured into 150 cc. concentrated NH4OH, stirred 0.5 hr., the dried CHCl3 layer evaporated, and the residue in EtOH treated with C and diluted with H2O gave 0.2 g. 2-chloro-4-methyl-1'-phenyltriazolo(4',5':5,6)quinol

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ne, m. 180° (from EtOH and petr. ether). IVa (5 g.), 3.5 g.
o-ClC₆H₄CO₂H, 4.5 g. K₂CO₃, and a little Cu bronze refluxed 3
hrs. with 30 cc. C₅H₁₁OH, the solvent steam-distd., the residue filtered,
acidified with HOAc, the ppt. dried, refluxed 2 hrs. with 12 cc. POCl₃,
dild. with 150 cc. CHCl₃, added to 300 g. ice and 50 cc. concd. NH₄OH,
stirred 0.5 hr., and the CHCl₃ layer dried and evapd., gave 1.5 g.
3-phenyl-11-chloro-3H-triazolo[4,5-c]acridine, purified in C₆H₆ on Al₂O₃, m.
219-21°, which changed above 200° to a polymorph, m.
229-30°.

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TITLE: Preparation of indolocarbazoles. VII. Some derivatives
of 5-amino-1-phenylbenzotriazole
AUTHOR(S): Carter, P. H.; Katritzky, A. R.; Plant, S. G. P.
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GI For diagram(s), see printed CA Issue.
AB Anthracene (I) and 1-nitronaphthalene (II) react at about 300° to
give as the principal product 7,12-dihydro-7,12-o-benzenbenz[a]anthracene
(III), the structure of which was established by an independent synthesis.
The mechanism of its formation was postulated to involve (1) an
synthesis: followed by the elimination of the elements of HNO₂, the latter
undergoing reduction to NO. Naphthalene (IV) has been condensed with II to
give 7,11-dihydro-7,14-o-benzenbenz[a]naphthalene (V). I and its 2-Me
derivative (VI) were condensed with 1,4-dihydronaphthalene (VII), and the
resulting adducts were dehydrogenated to yield 5,12-dihydro-5,12-o-
benzenonaphthalene (VIII) and its 2-Me derivative (IX), resp. A new
of 1,2-dihydronaphthalene (X) has been developed involving the reduction of
1-CIOH₂OH to the 1,2,3,4-tetrahydro derivative in alkaline medium and in the
presence of a Ni catalyst followed by the dehydrogenation of the
secondary alc. in the presence of HCl. II (8.8 g.) and 8.8 g. I
heated 15 h. under N at 300°, and the mixture distilled at 12-14 mm.
gave 7.62 g. distillate up to 177°, fraction A; the residue
dissolved in C₆H₆ and distilled further up to 250° and 1 mm. gave 3.82
g. distillate, fraction B; the residue from this distillation sublimed at
250° and 1 mm. gave 2.7 g. sublimate on the cold finger and 1.78 g.
final residue. Fraction A crystallized from C₆H₆ gave 2.74 g. I; the mother
liquor evaporated and the residue crystallized from ligroine gave II, light
yellow
needles. The combined fraction B and the sublimate worked up by solvent
fractionation from C₆H₆ and hexane, the crude product in hexane passed
through Al₂O₃, and the resulting white crystalline hydrocarbon (4.5 g.)
recrystd. from ligroine gave III, 208-9°. A solution of III and
picric acid in EtOAc slowly evaporated gave the picrate of III, m.
147-7.5°. III and 1,3,5-C₆H₃(NO₂)₃ in 1:1 EtOH-C₆H₆ gave an
adduct, m. 173-4°. III gave with 2,4,7-trinitrofluorenone (XI) in
C₆H₆-EtOH the III·XII adduct, deep red solid, m. 189.5-90.5°. III
(2.0 g.) in 30 cc. Ac₂O and 30 cc. glacial AcOH treated at about
10° with stirring with 0.6 cc. fuming HNO₃ (d. 1.50) in 15 cc.
AcOH, the mixture kept 0.5 h. at 0-2°, and then 2 h. at 25°,
the resulting yellow crystalline product washed with glacial AcOH and dried,
the mother liquor diluted with ice water, the residue digested with hexane,
and the combined ppts. recrystd. from Ac₂O gave 1.4 g. NO₂ derivative of
III,
yellow plates, m. 308-9°. III shaken with 10 cc. CHCl₃, and the
solution treated with a small amount acid-washed Fe powder and then dropwise
with Br in CHCl₃ (a few drops were sufficient to color the solution light
yellow) gave no decolorization even after 1.5-h. refluxing, but yielded
only recovered III. III (3 g.) in 15 cc. PhNO₂ added to 2.4 g. AlCl₃ in
15 cc. PhNO₂, the resulting red-brown solution treated at 10° with
swirling with 0.96 cc. Ac₂O, cooled 25 min., kept 3.5 h. at 20°,
and poured onto crushed ice and 15 cc. concentrated HCl, the mixture steam
distilled,
the residue cooled, the solid filtered, dissolved in ligroine, treated
with Norit, filtered, and cooled, and the white microcryst. material (1.34
g.) recrystd. from 1:1 CHCl₃-hexane gave an Ac derivative (XII) of III,
white
crystals. Crude XII (0.1 g.) and 0.1 g. NH₂OH·HCl in 5 cc. pyridine and 5
cc. EtOH refluxed 2 h., the solvent removed, the residue digested with
H₂O, and the crude material (0.1 g.) dried and recrystd. from C₆H₆ and
then from 2:1 C₆H₆-EtOH yielded the oxime of XII, white microcrystals, m.
273-5°. III (3 g.) in 40 cc. glacial AcOH treated dropwise during
0.5 h. on the steam bath with 2.64 g. CrO₃ in 25 cc. glacial AcOH, the
flask swept slowly 10 h. with N (to give 0.315 g. weight increase in an

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attached CO₂ absorber), the soln. dild. with 2 vol. ice water, and the
yellow flocculent solid (0.5 g.) recrystd. from 2:1 EtOH-C₆H₆ gave
anthraquinone, m. 275-6°. III (3 g.) and 1.5 g. 2:1 Cu
-Al₂O₃ catalyst heated 5.5 h. under 106 atm. initial H pressure
to 275° (max.), the mixt. evapd., the residue steam distd. to give
0.72 g. colorless oil, lighter than H₂O, and 0.44 g. light yellow oil,
heavier than H₂O, and to leave 1.19 g. greenish yellow viscous residue;
the 1st fraction crystd. partially on standing; fractional pptn. of
the picates yielded a small amt. of picate, m. 135-45° (not further
purified), which decompd. with H₂O yielded an amt. of white crystals only
sufficient for a m.p. detn., m. 78-9°. In a similar run with 0.2
g. III the product was distd. to give CIOH₂ and a 2nd blue-green
fluorescing fraction, which recrystd. from EtOH gave 0.04 g. white
material, m. 85-105°; this dehydrogenated with Pd-C at 350°
gave 1. m. 212°. I (10.4 g.), 8.76 g. 1,4-naphthoquinone, and 80
cc. PhNO₂ heated 24 h. at 140-50° and the mixt. dild. with 400 cc.
MeOH yielded 12.9 g. quinone (XIII), yellow solid, m. 275-7° (from
dioxane). XIII (3.2 g.), 4 g. NH₂OH·HCl, 25 cc. pyridine, and 25 cc. EtOH
refluxed 3 h., the solvent removed, the residue triturated with H₂O, the
solid (3.1 g.) filtered off, washed with H₂O, and dissolved in 30 cc.
glacial AcOH, and the soln. cooled deposited 1.1 g. monoxime (XIV), yellow
crystals, m. 233-6° (decompn.); the filtrate dild. with H₂O pptd.
1.46 g. dioxime, m. 178° (decompn.) (from aq. EtOH). XIV (0.5 g.)
in 20 cc. EtOH heated on the steam bath and treated with swirling with 2.2
g. SnCl₂ in 10 cc. concd. HCl, the soln. heated 3 min., cooled, dild. with
10 cc. concd. HCl, and cooled to 0°, and the white cryst. deposit
dissolved 1 h. at 105° yielded 0.06 g. crude amine HCl
salt; a 0.1-g. sample treated 0.5 h. with 3 cc. each Ac₂O and glacial
AcOH, the clear soln. dild. with H₂O, and the white ppt. (0.12 g.)
recrystd. from aq. AcOH yielded 6-acetamido-11-acetoxy-5,12-dihydro-5,12-o-
benzenonaphthalene, white microcrystals, m. 344-9° (decompn.).
Crude amine HCl salt (0.5 g.) suspended in 15 cc. glacial AcOH
at 10° and treated with stirring and cooling with 10 cc. concd.
H₂SO₄, 10 cc. AcOH, and 6 cc. H₂O, followed by 0.6 g. solid powd. KNO₂,
the mixt. stirred 1 h. in the cold, treated with 1 g. urea followed by 1.2
g. Na hypophosphite in 30 cc. concd. HCl, refrigerated overnight, dild.
with 100 cc. H₂O, and filtered, and the solid washed, dried, and recrystd.
with Norit A from C₆H₆-EtOH gave 0.34 g. XIV, yellow needles, m.
272-6°. VII (85% pure) (12 g.) and 13.8 g. I heated overnight in a
sealed tube at 242°, and the product fractionally crystd. from
C₆H₆, hexane, MeOH, and EtOH yielded 0.1 g. 5,5a,6,11,11a,12-hexahydro-
5,12-o-benzenonaphthalene (XV), m. 189-90°, a MeOH soln. evapd. and
steam distd. gave 0.96 g. brown oil and 4.36 g. white solid which yielded
4.54 g. picate of CIOH₂, m. 145-7°. XV (0.3 g.) and 0.1 g. Pd-C
heated 2 h. at 350° evolved 28.5 cc. H₂ and gave 0.21 g. pure VIII,
m. 256-7°. XV heated with Pd-C at 160° was not
dehydrogenated. XV (0.2 g.) heated with 0.33 g. chloranil at 140°
gave no VIII. 1-CIOH₂OH (22 g.) in 95% EtOH to make 140 cc. soln. treated
with 1 cc. 50% aq. KOH, the mixt. hydrogenated 1 h. at 1700 lb. initial
pressure at 82° over 2 g. Raney Ni, the EtOH distd. off, the
residue dissolved in Et₂O, the soln. washed with 24 cc. NaOH, dried, and
evapd., and the residual liq. distd. gave 12.3 g. X, b.p. 87-9°. X
gave in CS₂ with Br the dibromide, white prisms, m. 69-71°. VII
gave similarly a dibromide, white needles, m. 71-4°. I and X
heated 16 h. in a sealed tube at 230° or 18 h. at 250° gave
recovered I. Tetralin (104 g.), 400 cc. dry C₆H₆, and 77.3 g. o-MeC₆H₄(COCl
treated gradually with 119 g. AlCl₃, the mixt. heated 0.5 h. with stirring
and poured onto crushed ice and 125 cc. concd. HCl, the C₆H₆ layer steam
distd., and the residual oil fractionated gave 98 g. 5,6,7,8-tetrahydro-2-

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naphthyl o-tolyl ketone (XVI), yellow-green oil, b.p. 180-6°,
2,4-dinitrophenylhydrazones, bright red needles, m. 180-1° (from
EtOH). XVI (30 g.) refluxed 3 h. at 275° to give about 2 cc. H₂O,
the product cooled and distd., and the 1st fraction (12.6 g.), b.p. below
250°, partially dissolved in EtOH removed 8.6 g. recovered XVI; the
insol. residue recrystd. from ligroine gave 3.5 g. unidentified
yellow-green, fluorescent solid, m. 153-4°, picate, m.
136-9°, a 2nd fraction of the distn. (11.2 g.), b.p. 180-208°,
fractionally recrystd. from C₆H₆ and hexane yielded 1.2 g. white
hydrocarbon C₁₈H₁₄, m. 208-9°. The addn. compd. between the
isomeric 1,2,3,4-tetrahydrobenz[a] and b]-anthraquinones, m. 135°,
obtained by the method of Mikhailov and Chernova (C.A. 37, 3086.6)
refluxed 48 h. with Zn and aq. NaOH yielded 104
1,2,3,4-tetrahydronaphthalene, m. 219-25°, and 49%
1,2,3,4-tetrahydrobenz[a]anthracene (XVII), white plates, m.
102-4°. XVII (2 g.) and 1.5 g. p-benzoquinone refluxed 5 h. in 35
cc. EtOH, and the resulting yellow solid recrystd. from glacial AcOH gave
an adduct (XVIII), yellow crystals, m. 215-16°; it gave in the air
in light a white amorphous solid, m. above 300°, which was insol.
in alkali. XVIII (2.0 g.) in 80 cc. AcOH heated to boiling and treated
with 1 drop 40% HBr in AcOH, the mixt. heated 0.5 h. and dild. with
several vols. H₂O, and the white ppt. (2.0 g.) recrystd. from aq. AcOH
gave the corresponding hydroquinone, C₂₄H₂₀O₂ (XIX), m. 256-60°.
XIX (2.0 g.) in 75 cc. boiling AcOH treated dropwise with 0.43 g. KBrO₃ in
10 cc. H₂O, and the soln. cooled and dild. with 100 cc. H₂O yielded 2.0 g.
crude 1,2,3,4,7,12-hexahydro-7,12-o-benzo-8,11-benz[a]anthraquinone
(XX). Crude XX (0.33 g.) and 1.0 g. NH₂OH·HCl in 10 cc. EtOH refluxed 1
h., the mixt. treated with 1.5 cc. pyridine, refluxed again 1 h., dild.
with H₂O, and digested with 4N NaOH, the alk. soln. filtered and acidified
with HCl, and the yellow flocculent ppt. filtered, washed, dried (0.26
g.), and recrystd. from glacial AcOH gave the monoxime of XX, yellow
rosettes, decompn. above 230°. XX (2 g.) refluxed 2 h. with 25 cc.
EtOH and 2.0 g. NH₂OH·HCl and then 2.5 h. with 3 cc. pyridine, the mixt.
poured into H₂O, the greenish brown ppt. washed with H₂O and digested with
4N NaOH, the soln. filtered and acidified with HCl, the ppt. washed with
H₂O and dissolved in 20 cc. EtOH, the soln. warmed on the steam bath,
treated with 6.6 g. SnCl₂ in 30 cc. concd. HCl, heated 5 min., poured into
3 vols. H₂O, and filtered, the filtrate made strongly alk. with KOH, the
ppt. washed with H₂O and redissolved in dil. HCl, the soln. treated with
H₂S and filtered, the filtrate neutralized with NaOH, and the flocculent
ppt. washed with H₂O and dried gave 0.54 g. crude diamine;
a 0.3-g. sample in 25 cc. AcOH added dropwise with stirring to 0.4 g. NANO₂
in 40 cc. concd. H₂SO₄ and 40 cc. 50% H₃PO₂ at 0-5°, the mixt.
stirred 3 h. at 0°, treated with 100 g. crushed ice, and extd. with
C₆H₆, the ext. washed, dried, and evapd., the residue sublimed at
160-210° and 2 mm., the resulting yellow-white slightly gummy
material dissolved in 5 cc. C₆H₆, the soln. passed through Al₂O₃, the
column eluted with an addnl. 8 cc. C₆H₆, the clear, white slightly
fluorescent effluent evapd. to dryness, and the residue recrystd. from
hexane yielded 0.030 g. 1,2,3,4,7,12-hexahydro-7,12-o-benzenbenz[a]anthra-
cene (XXI), small white rosettes, m. 194-5°. XXI (28 mg.) and 12
mg. Pd-C heated 0.5 h. in a N stream at 160-300° (5 cc. gas was
evolved), the product dissolved in C₆H₆, the soln. filtered and evapd.,
the residue dissolved in 1 cc. hexane at 60°, and the soln. cooled
gave 23 mg. III, m. 208-10°, III·XI adduct, m. 185-6°. IV,
m. 228-30° (prepd. from 1,2,3,4-tetrahydro-6,11-naphthalenequinone
by heating with Zn dust, NaCl, and ZnCl₂ to 260°) (1.44
g.) and 1.5 g. II heated 10 h. at 275° evolved NO; the black,
pitch-like residue dissolved in C₆H₆, and the soln. passed through Al₂O₃

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 gave 0.28 g. V, white crystals, m. 214-15°. V (0.1 g.) in 5 cc. CGH6 treated with 5 cc. hot satd. alc. XI yielded 80% XI-V adduct, m. 191-2.5°. 2-Methylanthraquinone in PhMe heated with hot alkali and Zn dust gave 25% VI, m. 205-7°. VII (2.6 g.) and 2.7 g. VI heated 12 h. at 245° in a sealed tube, the product removed from the tube with CGH6, the CGH6 soln. evapd., and the residue fractionally recrystd. from ligroine and EtOH gave 0.75 g. 2-Me deriv. (XXII) of XV, white crystals, m. 165-7°. XXII (0.25 g.) heated 1 h. with Pd-C to 255° (max.) gave 30 cc. H₂ the residue recrystd. from abs. EtOH yielded 0.2 g. IX, white crystals, m. 140-65°. I and 2-ClOH7NO2 heated at 300° during 18 h. gave a considerable amt. of gas, but no NO, and only I was recovered from the black pitchy solid. I and PhNO2 heated 20 h. at 270-85° yielded 20% pinkish crystals, m. 251-3° (chromatographed on Al2O3 with CGH6 and hexane), with 91.3% C and 5.17% H; at 350° during 27 h. 21% unreacted I and 10% anthraquinone were the only isolable products; at 305-10° (48 h.), 13% I was recovered and about 1% white solid, m. 260-76°, with 93.1% C and 5.53% H. Phenanthrene and II heated 17 h. at 340° gave much unreacted phenanthrene and no NO. Chloroanthracene and II heated 18 h. at 350° evolved NO and HCl and gave "dichloroparaanthracene" (C14H9Cl2) 2, m. 235° IV and II heated together gave no NO but the mkt. turned black; no pure product was identified.

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 ORIGINAL REFERENCE NO.: 50:1720a-i, 1721a-i, 1722a-i, 1723a
 TITLE: The reaction of aromatic nitro compounds with polynuclear hydrocarbons at elevated temperatures
 AUTHOR(S): Hurd, Charles D.; Juel, Leslie H.
 CORPORATE SOURCE: Northwestern Univ., Evanston, IL
 SOURCE: Journal of the American Chemical Society (1955), 77, 501-6
 CODEN: JACSAT; ISSN: 0002-7863
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 218 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 AB Distilled VII (1 volume) in 3 vols. CGH6 or cyclohexane added below 30° to 2 vols. each of concentrated H2SO4 and H2O under N or CO2, agitation continued 5 min., and the upper layer separated and washed with a small amount of aqueous NaHCO3 gave 77-92% (by anal.) Me3CCH2CHMeCHO (X); distillation yielded X in 90-2% purity, b100 78°; 90% H3PO4 under the same conditions gave 92% X. The isomerization was also carried out continuously in an all-glass apparatus by countercurrent feeding of VII and dilute H2SO4 and gave 82% X. By either method aqueous HCl or p-MeCGH4SO3H gave inferior results. Distilled VII alone or in solution dripped slowly on 2% by volume of solid catalyst, and the whole stirred under N 0.25 h. at room temperature and 0.5 h. at 70°, neutralized, and distilled gave the following yields of X (catalyst, % concentration of VII in solvent, % yield): ZnCl2, no solvent, 4%; ZnCl2 25% in cyclohexane (XI), 7%; ZnCl2 12.5% in XI 84%; ZnCl2, 25% in CGH6 (XII), 84%; ZnCl2, 25% in IV, 88%; FeCl3, no solvent, 25%; FeCl3, 25% in XI, 91%; FeCl3, 12.5% in XI, 87.5%; FeCl3, 25% in XII, 87.5%; FeCl3, 25% in IV, 48%; HgCl2, 25% in XI, 5.5%; CoCl3, 25% in XI, 5%; SnBr4, 25% in XI, 78%; SnCl2, 25% in IV, 75%; BF3.Et2O, 25% in IV, 62%; TiCl4, 25% in XI, 66%; NaH2PO4, 25% in XI, 6%; Al2(SO4)3, 25% in XI, 8%; potash alum, 25% in XI, 4%; zeocarb H.I., 25% in XI, 8%; zeocarb H.I., 25% in XII, 22%; supercel, 25% in XI, 4%; fullers earth, 25% in XI, 58%. No isomerization of VII in XI occurred with NiCl2, HgCl2, or alumina (XIII). Vapor-phase isomerizations were studied in a Pyrex U-tube immersed in a fusible-metal bath. One arm, 15 mm. in diameter, was filled with granules of XIII (8-10 mesh) supported on a sintered glass plate (catalyst volume 12-18 mL), and VII was introduced into the other arm, which acted as a vaporizer and preheater. Maximum yields of 83-7% X were obtained at 280-340°, the yield was unchanged by contact time varying from 2 to 100 s. The catalyst life was >100 h. although it became covered with a thin film of C. Granulated acid-washed fullers earth (XIV), after an initial period of lower yields, gave a 98% yield; XIV was a less vigorous and more easily controlled catalyst. Pumice or kieselguhr impregnated with H3PO4 gave up to 90% XI with a diluent for the VII and high acid concns. and high feed rates avoided. Oxidized Al turnings gave a 76% yield. Of the vapor-phase catalysts, SO3 gave a 74% yield with considerable charring, BuH2PO4 10-12%; and SO2 3-4% at 250°. VII (128 g.) added dropwise with stirring below 60° to 200 g. Ac2O and 2 g. H2SO4, the whole kept 1 h., neutralized with an excess of anhydrous NaOAc, filtered, the filtrate concentrated in vacuo, the residue partitioned between Et2O and H2O, and the Et2O layer separated, washed with aqueous NaOH, concentrated, and distilled gave 22.1 g., b12 76° [shown to be impure Me3CCH2C(CH2)CH2OAc, hydrolysis giving VIIIb, b20 75-80°, n20D 1.4440 (3,5-dinitrobenzoate, m. 47-8°)], and 82.9 g., b12 114° [shown to consist of 15% Me3CCH2CMe(OAc)CH2OAc and 85% Me3CCH2CHMeCHO (XVI) (50 g.)] added slowly to 100 mL Ac2O and 2 g. H2SO4 gave 37.8 g. XV, b10 112-14°, n20D 1.4274. VII (100 g.) and 500 mL EtOH heated 5 h. at 220° and distilled gave much unchanged VII and then 15.7 g. VIIIb, b12 71-4°, n20D 1.4409 [p-nitrobenzoate, m. 30-5° (from alc. between 20° and -50°)]; acetate, b16 73°, n20D 1.4280; hydrogenation of VIIIb over Adam's catalyst gave Me3CCH2CHMeCHO (XVII). VII heated alone from 1.75 to 24 h. at 200-75° gave complex mxts. of products, the only compds. identified being VIIIb and XVII. VII (10 g.) in 20 mL medicinal paraffin heated 18 h. at 275° also gave a complex mixture in which only XVII could be identified. Me3CCH2CMeCHO (23.5 g.) and 63 mL 3N Al(OCMe2)3 in

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 AB ZnO, after activation by ions of heavy metals, appears to be a specific photocatalyst for reduction of methylene blue (I) and of thionine by CH2O, glycerides, or succinic acid. Ions of such heavy metals as Cu⁺, Pb²⁺, Bi³⁺, Tl⁺, Ag⁺, and Mn²⁺, enter the photocatalyst in the state of active centers and generate a specific distribution of energy levels, which favors photocatalysis. The active centers are labile formations with irregular structures that are not in equilibrium with their surroundings. They have a double structure: one portion serves for light absorption, the other for catalysis. The surface of such a photocatalyst has 3 zones: one where the ZnO shows the regular lattice, one where the ions of the activator are located, and one that surrounds these defects. The concentration of the heavy metals is critical; thus, changing the amount of Cu from 0.0255% to 0.00887% in slow steps changes the color of the luminescence from green to weak green to yellowish green to yellowish brown to yellow-orange to red-orange, and similarly changing the amount of Pb from 0.0626 to 0.00187% changes the luminescence from yellow-brown to red. The following materials have an extinguishing effect on the luminescence: KI, KBr, KCNS, K2CO3, K2S2O8, KClO3, KBrO3, KIO3, KMnO4, AcONa, Na2HPO4, Na2SO4, Na2F2, Na2S.9H2O, HCO2Na, NaCl, La acetate, BaCl2.2H2O, CaCl2.6H2O, Pb(OAc)2, CoCl2, MnSO4, ClCH2CO2H, BzOH, pyrogallol, rosolic, oxalic, sulfanilic, formic, adipic, and oleic acids, phenol, water, I, the leucobase of I in the presence of other reduction products of I, CH2O, methyl violet, hydrazine, hydroquinone, EtOH, and diphenylamine.

ACCESSION NUMBER: 1956:7209 CAPLUS
 DOCUMENT NUMBER: 50:7209
 ORIGINAL REFERENCE NO.: 50:143d-g
 TITLE: The nature of the composition of active centers in photocatalysis
 AUTHOR(S): Barshchevskii, I. N.; Nikolaev, L. A.
 CORPORATE SOURCE: Inst. Railroad Transport, Eng., Moscow
 SOURCE: Problemy Kinetiki i Kataliza, Akad. Nauk S.S.S.R. (1955), 8, 61-7
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 iso-PrOH heated under a fractionating column until the calcd. amt. of Me2CO was collected, and the mxt. distd. gave 13.0 g. Me3CCH2CMeCHO, b20 75-7°, n20D 1.4501; 3,5-dinitrobenzoate, flat needles, m. 80°. Similarly, 22.3 g. CH2=C(Me)CHO and 60 mL 3N Al(OCMe2)3 in iso-PrOH gave 7 g. VIIIb, b12 72-3.5°, n20D 1.4441; 3,5-dinitrobenzoate, m. 56°; 1-naphthylurethane, m. 83-5° (from petr. ether). Shaking IX 1 h. with 10% wt. aq. H2SO4 gave principally VIIIA (3,5-dinitrobenzoate, m. 120-1°), and small amts. of Me3CCH(OH)CMe2OH, m. 62-3°, and IIA (2,4-dinitrophenylhydrazone, orange-red, m. 154-6°). In the vapor phase at 279-90° over XIII, IX gave a complex mxt. of ketones in which Me3CCOCMe2 was identified, along with possibly Me3CCH2CMe2CO2H, m. 195-7°, and Me3CCMe2CHO, camphorlike solid, b12 46-52°, m. 109-19° (2,4-dinitrophenylhydrazone, yellow, m. 157-9°). IIA, synthesized by the Favorskii method (Favorskii and Fritzmann, J. Russ. Phys. Chem. Soc. 44, 1351(1912)), gave a 2,4-dinitrophenylhydrazone, orange-red leaflets, m. 161-2°. Crude XVI vaporized in a Cu coil, mixed with H (6-7 mol H/mol XVI), and passed over 290 g. Cu-kieselguhr pellets in a Pyrex tube 2.5 cm. in diam. at 200 ± 10° and a contact time of about 20 s. gave 60% XVII. The vapor pressure of XVII and the corresponding temp. were as follows: 20, 79.7°; 40, 93.1°; 60, 101.6°; 200, 131.2°; 400, 151.2°; and 760, 172.4°. (CH2)4(CO2H)2 (36.5 g.), 100 g. XVII, 19 g. XII, and 0.1 mL concd. H2SO4 refluxed 6 h. with the H2O removed continuously, the XII distd., and the residue washed and distd. gave the adipate, b1 178°, n20D 1.4452. The following esters of XVII were similarly prepd.: 2,4,4-trimethylvalerate, b1 92-3°, n20D 1.4307; fumarate, b2 168-72°, n20D 1.4540; maleate, b1 167°, n20D 1.4518; pimelate, b1 5 192°, n20D 1.4461; oleate, b1 208°, n20D 1.4547; and the 3-cyclohexene-1-carboxylate, b2 112°, n20D 1.4600. XVII (109 g.) added slowly to 38 g. POC13 in 107 g. XII, and the whole warmed until HCl evolution ceased, washed, and distd. gave the orthophosphate, b2 184°, 20D 1.4395. XVI (410 g.) decompd. to give mainly VIII; at 30-50°, was formed a more stable peroxide (this was demonstrated by peroxide titrn.) which was not decompd. to XVIII. XVIII (100 g.) and 70 mL SOCl2 warmed until HCl evolution ceased and then fractionated gave 107 g. acid chloride (XVIIIa), b750 146-5°. XVI (200 g.) and 500 mL 10N MeOH-NH3 hydrogenated 2 h. at 30 atm. and 120° over Raney Ni gave 173.2 g. Me3CCH2CHMeCH2NH2 (XIX), b78 79-84°, picrate, yellow plates, m. 190°; sulfate, colorless plates, m. 268-72° (from hot H2O on diln. with EtOH). XVI in 4 vols. 4N MeOH-NH3 and a 10-fold excess of H passed over 28% Ni on kieselguhr at 200° contact time 20 s., gave 45% XIX and 10% (Me3CCH2CHMeCH2)2NH (XX). The oily oxime from 10 g. XVI, 10 g. H2NOH.HCl, 15 g. KOAc, 100 mL MeOH, and 50 mL H2O dissolved in 500 mL abs. EtOH, added in 1 portion to 50 g. Na cut into small pieces, the reaction allowed to proceed vigorously with occasional cooling, the mxt. then heated to dissolve any unreacted Na, and the whole dild. with H2O gave 22% XIX, b12 45-7°. XIX (197 g., 88% pure), 105 g. XVI (85% pure), 150 mL MeOH, and Raney Ni hydrogenated at 50 atm. and 110-25° gave 154 g. XX, b1 121-4°, n20D 1.440; HCl salt, m. 134-7° (from

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 EtOAc). To 50 g. XX was added 50 mL. 98% HCO₂H, with the temp. kept below 40°, 50 mL. 40% aq. HCHO then added, the whole refluxed 2 h., poured into H₂O, made basic with NaOH, the oil which sepd. extd. into Et₂O, and the ext. concd. and distd. gave 34.8 g. (Me₃CHCH₂CHMeCH₂)₂NHMe (XXI), b₁₃ 127-1°, n_D 1.4377 (picrate, yellow plates, m. 128-9°; HCl salt, m. 238° (from aq. EtOH)); as byproducts were also formed Me₃CHCH₂CHMeCH₂NHMe₂ (XXIII), b₁₀ 47-50°, n_D 1.4220, and XVI, in approx. equal parts. XXI (2 g.) and 5 g. MeI heated 2 h. at 110° gave 1.35 g. methiodide, m. 198° (decompn.) (from aq. EtOH). XXI (20 g.), 16 g. MeBr, and 50 mL. EtOAc contg. a crystal of NaI heated 4 h. at 110° gave 18.9 g. methobromide, m. 209-10°. IIA (68 g.) in 150 mL. 10N MeOH-NH₃ hydrogenated over Raney Ni at 55 atm. and 120-40° gave 49% Me₃CHCH₂CHMeNH₂, b. 118-21°, n_D 1.416; picrate, yellow needles, m. 181-2°. XVI (400 g.) and 235 g. Et₂NH in 200 mL. MeOH hydrogenated over Raney Ni at 50 atm. and 100° gave 16% Me₃CHCH₂CHMeCH₂NH₂ (XXIII), b₁₀ 70-1°, n_D 1.4272, b₁₀ 10-4° (picrate, m. 96-7° (from EtOH)); the higher-boiling material was shown to contain 7 g. Me₃CHCH₂CHMeCH(OH)NH₂ (XXIV), b₁₀ 90-100° (picrate, m. 104-5°). XXIII gave a methiodide, m. 101-2° (from Me₂CO-Et₂O), and a methobromide, hygroscopic, m. 110° (from Me₂CO-EtOAc). Similarly XVI and Me₂NH gave XXII, b₁₁ 53-63°, n_D 1.423; picrate, m. 105-96° (sic) (from EtOH); HCl salt, m. 167-8° (from iso-PrOH-Et₂O). XXII gave a methiodide, m. 238° (decompn.) (from Me₂CO-EtOAc) and a methobromide, m. 275° (decompn.) (from EtOH-EtOAc).
 ACCESSION NUMBER: 1955:73372 CAPLUS
 DOCUMENT NUMBER: 49:73372
 ORIGINAL REFERENCE NO.: 49:13881c-1,13882a-1,13883a-c
 TITLE: Oxidation products of diisobutylene. II. Isomerization of 1,2-epoxy-2,4,4-trimethylpentane, and some products derived from 2,4,4-trimethylpentanal
 Gasson, E. J.; Graham, A. R.; Millidge, A. F.; Robson, I. K. M.; Webster, W.; Wild, A. M.; Young, D. P.
 Distillers Co. Ltd., Epsom, UK
 SOURCE: Journal of the Chemical Society, Abstracts (1954) 2170-9
 CODEN: JCSAAZ; ISSN: 0590-9791
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 and dried, gave 50 mg. thiosemicarbazide (III), from aq. MeOH (1:1), m. 147-51°; 10% HCl converted III to a difficultly sol. HCl salt; III (clear violet FeCl₃ color) in 50% MeOH with AgNO₃ gave a weak yellow color in the cold, and pptd. Ag₂S when heated or kept a long time in the cold. In the Angeli-Rimini test (15 min. at 20° with benzenesulfhydroxamic acid) II gave the same reactions as BzH: the alk. soln. acidified and treated with FeCl₃ gave a deep wine red color. II (300 mg.), 70 cc. 10% HCl, and 80 mg. PtO₂ with H after 40 hrs. absorbed 2 moles H; 15% II was the only cryst. product obtained. II (80 mg.) refluxed 40 min. with 27 mg. MeNH₂.HCl in 3 cc. alc., concd. in vacuo, the residue rubbed and treated with ether gave 64 mg. (probable) N-(4-dimethylamino-2,3-bis(p-hydroxyphenyl)-1-butylidene) methylaniline-HCl (IV), m. 202-6° (decompn.) (with an addnl. 8 mg., m. 189-92° (decompn.) from the mother liquor); IV, sol. in H₂O and alc., insol. in ether, Me₂CO, was decompd. on attempted recrystn. from alk.-ether; IV heated in alc. gave off MeNH₂ (odor, litmus turned blue); this soln. did. with ether gave about 50% recovery of material with an unsharp m.p. IV with aq. NaHCO₃ gave II as the free base, m. 212-17° (decompn.). IV (95 mg., prepd. from II with MeNH₂.HCl and used without isolation) in 10 cc. alc. with catalyst (from 20 mg. PtO₂ prehydrogenated in 5 cc. alc.) and H (1 mole H was absorbed in 1 hr.; absorption then tapered off but never stopped) the product filtered, and the filtrate treated with ether to give a turbidity, yielded 41 mg. 4-dimethylamino-2,3-bis(p-hydroxyphenyl)-1-methylaminobutane-HCl (V), m. 206-15° (decompn.), with an addnl. 18 mg., m. 200-8° (decompn.) (neg. Angeli-Rimini color reaction) (green-blue FeCl₃ color). V in alc. treated with HCl in ether gave the di-HCl salt, m. 243-8° (decompn.). V (23 mg.) in 10 cc. H₂O with about 50 mg. NaHCO₃ extd. with EtOAc, the ext. concd., the free base taken up in 5 cc. MeOH and heated 12 hrs. at 100° with 5.5 mg. NaHCO₃ and 1 cc. MeI gave 18 mg. crude product, m. 225-40° which was taken up in alc. and pptd. with ether many times to give [CH(C₆H₄OH-p)CH₂NHMe]₂ (VI), m. 259-62° (decompn.) (leaflets, m. 300° formed in the melt). II (200 mg.) was refluxed 3 hrs. with 110 mg. Me₂NH.HCl in 20 cc. alc., the soln. filtered, and the filtrate acidified with 1 drop ether-HCl, crystd. with ether and Me₂CO, and afforded 104 mg. [CH(C₆H₄OH-p)CH₂NHMe₂.HCl]₂ (VII), m. 257-67° (decompn.) (green-blue FeCl₃ color). VII (50 mg.) refluxed 1.5 hrs. with 1 cc. HCl-satd. HOAc and 1 cc. AcCl, kept overnight, concd. to dryness, the residue treated with ice cold aq. NaHCO₃ and concd. gave 35 mg. faintly yellow oil, the O-Ac base (VIII), which was distd. in a bulb tube and gave 22 mg. colorless oil, b_{0.02} 200-10°; VIII in 2 cc. alc. kept overnight at room temp. with 2 drops concd. HCl regenerated VII, from alc.-ether, m. 263-7° (decompn.). VII (48 mg.) in 0.5 cc. H₂O with 50 mg. NaHCO₃ (kept a long time in the cold, and rubbed from time to time, gave no pptn. of the free phenolic base) extd. with EtOAc, the ext. dried (Na₂SO₄), concd. to dryness, the colorless resin taken up in 10 cc. MeOH and heated 4 hrs. at 100° with 1 cc. MeI gave 22 mg. crude VI, m. 253-63° (decompn.); VI taken up many times in alc. and pptd. with ether, microscopic tufts of needles, m. 262-4° (decompn.), with the melt forming leaflets, m. 300-10°.
 ACCESSION NUMBER: 1955:39389 CAPLUS
 DOCUMENT NUMBER: 49:39389
 ORIGINAL REFERENCE NO.: 49:7533e-1,7534a-1,7535a
 TITLE: New compounds with the basic skeleton of synthetic estrogens. V. Formation of 4-dimethylamino-2,3-bis(p-hydroxyphenyl)-1-butanal from dimethylaminomethyl-p-

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 AB p-AcOCH₂CH₂CH₂NHMe₂ (I) in C₆H₆ shaken with H₂ at room temperature or on a water bath, or with Ag, Zn powder, or Cu-bronze on a water bath gave no coupling; only I or dimer could be recovered. I (7.1 g.) in 200 cc. PhMe (from 12 g. I.HCl) was refluxed 70 min. with 8 g. Na with protection from moisture (when the solution gave no test for Cl⁻ ion), the mixture filtered and concentrated, and the residue (4.38 g., with an alkaline reaction to litmus paper) was taken up in 30 cc. MeOH and allowed to stand many days in a stoppered flask (an amine odor was detected), the yellow precipitate was filtered off, washed with MeOH and ether and dried at 100° in vacuo, to give 1.76 g. crude 4-dimethylamino-2,3-bis(p-hydroxyphenyl)butanal (II), m. 206-9° (decomposition), which was taken up in 20 cc. H₂O and several drops of HOAc or HCl, treated with charcoal, and neutralized with K₂CO₃, and the yellow precipitate refluxed 2 hrs. in 100 cc. MeOH, and cooled, giving 1.3 g. II, m. 216-17° (decomposition); for further purification, 670 mg. was refluxed in 1200 cc. 96% alc. to give complete solution, the mixture concentrated in vacuo to about 350 cc. and cooled 2 hrs. at 0°, to give 290 mg. II, m. 217-18° (decomposition). II (137 mg.) (violet FeCl₃ color) in excess 0.1N HCl, back titrated with 0.1N NaOH (to methyl red) using 4.485 cc. 0.1N HCl. II (136.2 mg.) in excess HCl diluted with 300 cc. H₂O and back-titrated potentiometrically with 0.1N NaOH using 4.38 cc. 0.1N HCl. I (3.6 g.) in 90 cc. PhMe (from 5.4 g. I.HCl) was refluxed with 5 g. Na, with a stream of N passing through the apparatus and into 5 cc. 2N HCl; the reaction was worked up as before and gave 530 mg. crude II; the acid solution was titrated to methyl orange with 0.1N NaOH: 2.75 cc. 0.1N HCl needed corresponded to 12.4 mg. Me₂NH. II in excess 10% HCl refluxed 2 hrs., neutralized with K₂CO₃ gave II. II refluxed 4 hrs. in excess 10% NaOH gave some decomposition with formation of a gaseous amine; neutralization with CO₂ regenerated only 37% II. II distilled in a bulb tube gave at 180°/0.2 mm. decomposition of II and at 210-20° distillation of a colored oil which solidified as a glass and was no longer acid-soluble but alkali-soluble II with HCl, picric acid, or H₂PtCl₅ gave amorphous ppts., II with MeI gave an amorphous H₂O-soluble product. II (250 mg.), C₆H₅SH, and Ac₂O gave 80% diacetate, faintly colored oil, which was distilled without decomposition at 180-90°/0.003 mm. To 100 mg. II in 3 cc. H₂O and 0.3 cc. 2N NaOH was added (3 hrs.) with stirring 0.32 g. Me₂SO₄ and 0.9 cc. 2N NaOH, the product extracted in EtOAc was subjected to Hofmann's degradation by heating 2 hrs. with 20 cc. concentrated KOH (cleavage of the amine started in the cold); the amine was identified as Me₃NHCl; the neutral olefin [22 mg., pos. reaction in CCl₄ to C(NO₂)₄] was distilled in a bulb tube at 170-85°/0.25 mm. but did not crystallize; a (nonvolatile) basic portion [27 mg., neg. C(NO₂)₄ reaction] did not crystallize. II (110 mg.) methylated with Me₂SO₄ was taken up in 1.5 cc. HOAc, added to 56 mg. CrO₃ in 0.75 cc. HOAc, heated 25 min. on a steam bath, diluted with H₂O, extracted with ether, and the product from the ether extract was distilled in a bulb tube at 0.5 mm. to give anisic acid, subliming at 100-40°, m. 168-77°, and anisil, subliming at 210°, m. 121-6° (about 10% yield). To 50 mg. II in 0.5 cc. 3% HOAc was added 50 mg. H₂NCSNH₂.HCl in 2 cc. H₂O and the mixture allowed to stand overnight, neutralized with K₂CO₃, and the derivative which separated washed with cold H₂O

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 acetoxyphephenylchloromethane
 AUTHOR(S): Bretschneider, H.; Sachsenmaier, W.
 CORPORATE SOURCE: Univ. Innsbruck, Austria
 SOURCE: Monatshefte fuer Chemie (1953), 84, 629-39
 CODEN: MOCHB7; ISSN: 0026-9247
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 AB The known process of preparing H₂N(CH₂)₆NH₂ (I) by treating NC(CH₂)₄CN (II) with H under pressure and at elevated temperature in the presence of Ni- and Co-containing catalysts and possibly of NH₃ and aromatic hydrocarbons, is improved by using Ni and (or) Co containing mixts. (or reaction products) with oxides of trivalent metals as catalysts, whereby the stoichiometric ratio of these compds. should correspond to the spinel type. Suitable catalysts of the formula XII₁₀X₂III₁₀O₃, where XII and XIII mean the stoichiometric equivalent of 1 or several bi- or trivalent metals, are prepared by mixing Al, Fe, and (or) Cr nitrate with Ni and (or) Co nitrate which may be partially replaced by equivalent amts. of Zn, Cu, Mg, and other metal nitrates, applying the mixture on silica gel, converting the products thus obtained to the oxides by calcining at about 700-80° and finally reducing at about 400° with H. Thus, silica gel 900 is impregnated with a solution of Co(NO₃)₂·6H₂O 582 and Ni(NO₃)₂·6H₂O 291 in water 360 parts, dried at 120° calcined at 750°, and reduced 2 hrs. in a current of H at 400°, the resulting catalyst 25 heated in a stirring autoclave with II 200, PhMe 200, and liquid NH₃ 200 parts at 85° under 300 atmospheric H until the H absorption is finished, the mixture cooled, the catalyst filtered off, and the filtrate distilled in vacuo to give PhMe as first runnings and pure I, snow-white crystals, f.p. 40° (distillation residue 3.1% output 96.5%).

ACCESSION NUMBER: 1953:66106 CAPLUS
 DOCUMENT NUMBER: 47:66106
 ORIGINAL REFERENCE NO.: 47:11230a-d
 TITLE: Hexamethylenediamine
 INVENTOR(S): Adam, Karl; Trieschmann, Hans G.
 PATENT ASSIGNEE(S): Badische Anilin- & Soda-Fabrik (I. G. Farbenindustrie Akt.-Ges. "In Auflösung")
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 848498		19520904	DE	

L20 ANSWER 222 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB Secondary amines are produced almost exclusively when the reaction of aromatic amines with alcs. is conducted over an alumina-supported catalyst containing 2-20% Cu and an oxide of Ca, Zn, Cr, Mg, Fe, or Mn (0.2-2.0 moles/mole Cu). PhNH₂, MeOH, and H in a molar ratio of 1:1.5:2.5 were passed 125 hrs. over 50 cc. of catalyst (4.6% Cu and 9.0% Zn, present as oxide) at 250° and 1 atmospheric at a liquid hourly space velocity of 0.89 to give a washed and dried product consisting of 2.8% PhNH₂, 96.1% PhNHMe, and 1.1% PhNHMe₂·PhNH₂ (96.8%) was converted to methylated products.

ACCESSION NUMBER: 1952:42397 CAPLUS
 DOCUMENT NUMBER: 46:42397
 ORIGINAL REFERENCE NO.: 46:7119a-c
 TITLE: Secondary aromatic amines
 INVENTOR(S): Deahl, Thomas J.; Stross, Fred H.; Taylor, Marion D.
 PATENT ASSIGNEE(S): Shell Development Co.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2580284		19511225	US	

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 AB Metal molybdates soluble in excess NH₄OH (I) are hydrogenation catalysts. Thus Ni molybdate was prepared as follows: To (NH₄)₆Mo₇O₂₄·4H₂O 1769 g. in distilled H₂O 5000 neutralized by 28% I 900 cc. was added with stirring to Ni(NO₃)₂·6H₂O 2908 in 5000 g. H₂O, the resulting slurry (pH 4) adjusted to pH 7 with I 500 cc., and the precipitate washed, filtered, dried, calcined 18 hrs. at 400° (it then contained 23.2% Ni, 46.4% Mo), and reduced in H with the temperature gradually increased for 7 hrs. to 550° and held 18 hrs. at 550°; the product contained 26.6% Ni and 53.1% Mo, corresponding to NiMoO₂·0.1 Mo₂O₃ (II). The following hydrogenations were carried out with 2-5 weight-% II. Cyclohexene, 100-150°, 135 atmospheric pressure, 100% yield; diisobutylene, 250°, 560 atmospheric, 85%; cyclopentadiene, vapor phase, 430-70°, 1 atmospheric, 40% cyclopentene; MeCO, 80-150°, 120-35 atmospheric, 100% Me₂CHOH; tetrahydrofuran, 300°, 560 atmospheric, 14% n-C₄H₁₀, 14% BuOH; MeCO₂Et (III), 250°, 560 atmospheric, 11.8% EtOH, 6.6% Et₂O; PhNO₂, 80°, 150 atmospheric, 100% PhNH₂; PhCN, 100-50°, 135-200 atmospheric, 23% PhCH₂NH₂, 9% 2,4,5-triphenylimidazole; EtCN, 125°, 200 atmospheric, 66% Pr₂NH, 33% PrNH₂; MeCO₂H, 200-50°, 700-1000 atmospheric, 55% III; C₆H₆, 150-75°, 150-200 atmospheric, 100% C₆H₁₂. Effective catalysts are also ZnMoO₃, CoMoO₂, CuMoO₃, and CoMoO₃. Cf. C.A. 46, 3226i, 3564c.

ACCESSION NUMBER: 1952:45376 CAPLUS
 DOCUMENT NUMBER: 46:45376
 ORIGINAL REFERENCE NO.: 46:7579h-i, 7580a-b
 TITLE: Metal molybdate hydrogenation catalysts
 INVENTOR(S): Arnold, Herrick R.; Carnahan, James E.
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2584531		19520205	US	

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 AB cf. following abstract An investigation of catalytic vapor-phase reactions of aromatic amines, particularly their alkylation with alcs., was undertaken at 360° to explore the advantages of continuous low-pressure reactions over high-pressure batch syntheses, such as that employed for PhNHMe₂. A study was then made, with the most active catalyst found, of certain process variables, including an extension of the results to higher alcs. In general the catalysts were prepared from reagent-grade chemicals by standard methods. Precipitated catalysts were washed free of salts, dried, and activated by heating with N 1 hr. at 360°. The catalysts employed included: lump Al₂O₃ dried at 50°, lump Al₂O₃ dried at 110°, powdered Al₂O₃, Al₂O₃ tablets, Al₂O₃ on asbestos, Al₂O₃ on pumice, H₃PO₄ on asbestos, H₃PO₄ on charcoal, Al₂O₃ with W₂O₃ on asbestos, Al₂O₃ with Cu, AlPO₄ on asbestos, TiO₂, Al₂(SO₄)₃ on asbestos, kaolin on asbestos, W₂O₃ with H₃PO₄ on asbestos, W₂O₃, Ca₃(PO₄)₂ on asbestos, silica gel, silica gel with Fe₂O₃, ZnO, CaSO₄, ThO₂, Cr₂O₃, and MgO. Of the many catalysts used, lump Al₂O₃ and ThO₂ and H₃PO₄ were the most active. While Al₂O₃ was the most efficient for N alkylation, all these catalysts effected more nuclear alkylation than had been anticipated. This led to a study of the rearrangement of alkyl groups from N to C, which was found to be accompanied by extensive disproportionation. The apparatus used consisted of a container for supplying the reactants, the furnace containing the reaction tube, and the apparatus for collecting the products of reaction. A mixture (50 ml.), containing 5 moles MeOH in 1 mole PhNH₂, was fed into the reaction tube at 0.8 ml./min. and the products collected and analyzed. A weighed sample was treated with Ac₂O at 100°, the excess Ac₂O hydrolyzed, the AcOH formed titrated with Ba(OH)₂, and amines calculated as PhNHMe. A temperature of 350-60° gave maximum conversion to PhNHMe₂. Methylation of o- and p-toluidine over Al₂O₃ at 360° gave as much as 90% tertiary amines. Alkylation expts. with PhOH and BuOH indicated that little tertiary amine was formed. Although conversions of primary to tertiary amines exceeding 90% were obtained, more effective catalysts must be found to permit the vapor-phase preparation of N,N-dialkylanilines in a reasonable degree of purity. Means were found for the syntheses from PhNH₂ of a wide variety of aromatic amines, including toluidines, xylylides, mesidine, and Ph₂NH.

ACCESSION NUMBER: 1952:8495 CAPLUS
 DOCUMENT NUMBER: 46:8495
 ORIGINAL REFERENCE NO.: 46:1471h-i, 1472h-i, 1473a-b
 TITLE: Catalytic reactions of aromatic amines
 AUTHOR(S): Hill, A. G.; Shipp, J. H.; Hill, A. J.
 CORPORATE SOURCE: Yale Univ.
 SOURCE: Journal of Industrial and Engineering Chemistry (Washington, D. C.) (1951), 43, 1579-83
 CODEN: JIECAD; ISSN: 0095-9014
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 224 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The preparation of (CH₂)_n (I), (MeCH₂)_n (II), and (CD₂)_n (III) is described, and infrared absorption spectra are shown for I and III. Zn dust (6 g.) was added to a stirred solution of 25 g. CuSO₄.5H₂O in 250 ml. H₂O, the mixture stirred 1 hr., the precipitated Cu allowed to settle, the supernatant liquid decanted, and the residue washed with H₂O, MeOH, and Et₂O; CH₂NH₂ prepared from 30 g. MeN(NO)CONH₂ was added over a period of 2 hrs. to a suspension of the precipitated Cu (IV) in a little Et₂O, the mixture allowed to stand 1 day, the ether solution decanted and discarded, the residue digested on the steam bath for several hrs. with 40 ml. 25% HNO₃, and the white flocculent amorphous precipitate filtered, washed with hot H₂O and then EtOH-H₂O (1:1), giving 0.7-0.8 g. I, m. 127-8°; a Cu-Ag alloy, Raney Ni, Naturkupfer C, and precipitated Ag were less satisfactory than IV as catalysts in the polymerization. A solution of MeCHN₂, prepared in 50% yield by the rapid addition of 30 g. EtN(NO)CONH₂ to a stirred mixture of 300 ml. Et₂O and 90 ml. 50% KOH at -15°, added dropwise to a suspension of IV in Et₂O gave 33% II, m. 94-6°. MeNO₂ (25 ml.) and 25 ml. 0.02 M NaOH in D₂O heated 24 hrs. at 110° in a sealed tube placed in a rocker type of shaker, cooled, and the lower layer dried over P₂O₅ and distilled gave 22.0 g. CD₃NO₂, d₂₅ 1.1672; 2 more exchanges with D₂O gave 17 g. (59%) CD₃NO₂ (V), d₂₅ 1.1832, practically free from H. DCl (VI) was generated from BzCl and D₂O and absorbed in a known weight of D₂O; a mixture of 6 g. V and D₂O (containing sufficient VI to neutralize the CD₃NH₂ formed) treated with D₂ under 3 atmospheric in the presence of 0.4 g. Pd-C, filtered under pressure through a porous glass disk, and evaporated to dryness in vacuo gave 5.9 g. (90%) CD₃ND₂.DCl, which on crystallization from BuOH m. 227-8°; the BuOH contained appreciable amts. of BuOD formed by the exchange reaction CD₃ND₂.DCl + 3BuOH → d₃l₃arw. CD₃NH₂.HCl + 3BuOD. CD₃NH₂.HCl (23 g.), 130 ml. H₂O, and 18.4 g. KCNO boiled gently for 15 min., and the CD₃NHCONH₂ thus obtained added slowly to a stirred solution of 23 g. H₂SO₄ in 150 ml. H₂O maintained at 0° gave 23 g. (60%) CD₃N(NO)CONH₂ (VII). CD₂NH₂ (3 g.), prepared either from VII and NaOH in D₂O (5 g. Na in 30 ml. D₂O) or from CH₂N₂ and D₂O, with an equal weight of IV gave 0.53 g. (53%) III, m. 122-3°; analytical data indicated the presence of residual H in III.

ACCESSION NUMBER: 1951:13733 CAPLUS
 DOCUMENT NUMBER: 45:13733
 ORIGINAL REFERENCE NO.: 45:2415d-i
 TITLE: Synthesis of organic deuterium compounds. I. Deutero-polymethylene
 AUTHOR(S): Leitch, Leonard C.; Gagnon, Paul E.; Cambron, Adrien
 CORPORATE SOURCE: Natl. Research Labs., Ottawa
 SOURCE: Can. J. Research (1950), 28B, 256-63
 DOCUMENT TYPE: Journal
 LANGUAGE: English

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 AB cf. C.A. 41, 2400d. A study of the conditions under which aromatic diazo compds. react with K₄Fe(CN)₆ (I) to yield arylazodiaryls shows that the decomposition proceeds most smoothly at pH 4-5 in the presence of AcONa buffer. Yields of about 20% can be obtained with R₂CH₂NH₂ when R is Me or Cl, but when R is MeO or NO₂, the yield is less than 0.5%. When R occupies the para position to the N in the original amine, the reaction product is the 2-aryl derivative of a 4,4'-substituted azobenzene; when R is an ortho substituent, the product is a 4-aryl derivative of a 2,2'-substituted azobenzene. Isomeric reaction products do not appear to be formed in appreciable quantity. PhNH₂ (23 g.) in 82 ml. HCl and 50 ml. H₂O, diazotized at 0-5° with 18 g. NaNO₂ in 50 ml. H₂O, the solution buffered with 100 g. AcONa in 200 mL, added slowly to 125 g. I in 600 ml. H₂O at room temperature, and the mixture kept overnight, gives 224 4-PhCH₂NH₂NPh, orange-yellow, m. 152°; if the precipitate which forms is separated immediately, it decompose to a black tar. The aqueous filtrate yields a little (PhN₂)₂ but no PhOH. Without AcONa, the yield is about 1% neutralization with NaOH or NaHCO₃ gives very tarry products. o-MeC₆H₄NH₂ (1 mol.) in 540 mL ice-cold 20% HCl, diazotized at 0-5° with 75 g. NaNO₂ in 200 mL. H₂O, the solution buffered with 272 g. AcONa.3H₂O in 225 mL. H₂O, slowly added to 840 g. I in 3 l. H₂O, and kept overnight, give 224 4-(o-tolyl)-2,2'-azotoluene (II), orange-red, m. 105.5°; the mother liquors from 5 mols. o-MeC₆H₄NH₂ yield 0.5 g. o-MeC₆H₄CN and 5 g. (2-MeC₆H₄N)₂. II (20 g.) in 500 mL boiling EtOH, added to 112 g. SnCl₂.2H₂O in 112 mL. concentrated HCl at 100°, gives 2.5 g. p-MeC₆H₄NH₂ and 7.1 g. 4-amino-3,2'-bitolyl (III), pale yellow viscous oil, b₀.005 92°, darkens rapidly on storage (Ac derivative (IV), m. 143°; di-Bz derivative, m. 140.5°; picrate, yellow, m. 198°). III (2 g.) in 50 mL. AcOH containing 2.5 g. AcONa, treated with 3% Br-AcOH, gives 5-bromo-4-amino-3,2'-bitolyl (purple oil), analyzed as the HCl salt, m. 160° (decomposition); di-Bz derivative, m. 175°. IV (10.5 g.) in 50 mL. AcOH at 25-30°, treated with 7 g. Cu(NO₃)₂.6H₂O and the pale yellow 5-NO₂ derivative (90%) hydrolyzed by refluxing 6 h. with 30% H₂SO₄ in EtOH, gives 5-nitro-4-amino-3,2'-bitolyl (V), orange, m. 95°. Reduction of V with Zn in boiling AcOH and the purple solution added to phenanthrenequinone in hot AcOH and heated 1 h. give 12-(o-tolyl)-10-methylphenanthrazine [8-(o-tolyl)-6-methyl-1,2,3,4-dibenzophenazine], light brown, m. 192-3°; brilliant purple solution in concentrated H₂SO₄. By a similar procedure, p-H₂CN₂CH₄NH₂ yields 10% 2-(p-tolyl)-4,4'-azotoluene (VI), m. 118°. Reduction of VI with SnCl₂ in alc. HCl gives 2-amino-5,4'-bitolyl (VII), b₀.4 123-8° [Ac derivative (VIII), m. 104° Bz derivative, m. 122°]; VIII and concentrated HNO₃ (20 min.), give 2',3'-dinitro-2-acetamido-5,4'-bitolyl(?) yellow, m. 191°. VII (1 g.) in 20 mL. CCl₄ containing a little AcONa, treated with 1% Br in CCl₄, gives the 3-Br derivative, m. 68° (Ac derivative, m. 135°; mono-Bz derivative, m. 195°). VIII (1.3 g.) and 3 g. POCl₃ in 15 mL. PhNO₂ (drop of SnCl₄ as catalyst), refluxed 4 h., give 60% 2,6,8-trimethylphenanthridine (C.A. numbering), pale yellow, m. 123° (HCl salt, pale yellow, does not m. at 250°). p-ClC₆H₄NH₂ (42.5 g.) in 150 mL. 25% HCl, diazotized below 5°, buffered with 91 g. AcONa in 55 mL. H₂O, and slowly added to 280 g. I in 1 l. H₂O (immediate evolution of N), and kept overnight, gives 13% 4,4'-dichloro-2-(p-chlorophenyl)azobenzene (IX), pale yellow, m. 153°; the residue yields 1% (4-ClC₆H₄N)₂ and some p-ClC₆H₄CN. Reduction of 10 g. IX with 15 g. Zn in boiling AcOH (with final addition of 5 mL. HCl in 20 mL. AcOH) gives 0.8 g. p-ClC₆H₄NH₂ and, after

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 AB Diazotization of o-aminobenzoylarylamines results in triazole condensation instead of Pschorr cyclization. If the imido H of an anilido compound is first substituted by radicals of great mobility, such as benzyl, and then diazotized, Pschorr cyclization will occur, giving an N-benzylphenanthridine. This with PC15 gives a chlorophenanthridine with elimination of the benzyl radical. Hydrogenation of PhCH₂NPh with Raney-Ni as a catalyst gives 80% PhCH₂NHPh which with o-O₂NC₆H₄COCl by the Schotten-Baumann method gives 81.6% 1-(o-nitrobenzoyl)-N-benzylaniline, pale yellow prisms, m. 108°. Reduction of this in HCl-alc. with SnCl₂ gives 93.4% N-(o-aminobenzoyl)-N-benzylaniline, white rectangular crystals, m. 107°. This is diazotized in the usual manner and the solution either shaken with Gattermann Cu or heated to 70° to give 54% 5-benzyl-6(5H)-phenanthridone (C.A. numbering), rhombic plates, m. 181°, which with POCl₃-PC15 yields 6-chlorophenanthridine (II), m. 117-18°. Identical with a compound obtained from POCl₃ on phenanthridone. Hydrogenation of I with PdO-CaCO₃ as a catalyst gives 60% phenanthridine, identical with a compound obtained by Zn-powder dry-distillation of phenanthridone. By the same method, the following were synthesized: 2-Ethoxy-5-benzyl-6(5H)-phenanthridone, colorless, long needles, m. 156-7°; 6-chloro-2-ethoxyphenanthridine, colorless, short needles, m. 160-2°; 2-ethoxyphenanthridine, white needles, m. 95-7°; 1(or 3)-methoxy-5-benzyl-6(5H)-phenanthridone, colorless needles, m. 152-4°.

ACCESSION NUMBER: 1951:3614 CAPLUS
 DOCUMENT NUMBER: 45:3614
 ORIGINAL REFERENCE NO.: 45:628f-i, 629a
 TITLE: Phenanthridine derivatives. II. A new synthetic method for phenanthridine
 AUTHOR(S): Mitsunashi, Kenmotou
 CORPORATE SOURCE: Toyama Coll. Pharm
 SOURCE: Yakugaku Zasshi (1943), 63, 177-82
 CODEN: YKXZAJ; ISSN: 0031-6903
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 fractional crystals of the Bz derivs. from aq. EtOH, 2 g. 4,5'-dichloro-2-benzamidobiphenyl (X), m. 191°. X (1 g.) and 3 g. POCl₃ in 10 mL. PhNO₂ contg. a little SnCl₄, refluxed 6 h., treated with 1 mL. POCl₃, and refluxed an addnl. 10 h., give 50% 2,8-dichloro-6-phenylphenanthridine, m. 193°, absorption max. at 2600, 3410, and 3575 Å.; picrate, mustard-yellow, m. 220°. p-O₂NC₆H₄NH₂ yields 0.25% 4,4'-dinitro-2-(p-nitrophenyl)azobenzene, brick-red, m. 240° (decompn.). p-MeOC₆H₄NH₂ gives 0.51 (4-MeOC₆H₄N)₂ and 0.25% p-MeOC₆H₄CN. 1-ClOH₂NH₂ gives 9% 4-(1-naphthyl)-1,1'-azonaphthalene (XI), brick-red, m. 178°. 4-Nitro-1,1'-binaphthalene (10 g.) in 20 mL. boiling EtOH, refluxed 1 h. with 45 g. 2nCl₂ in 40 mL. concd. HCl, the EtOH removed, and the residue poured into 300 mL. 30% NaOH, gives 8 g. 4-amino analog (XII), pink, m. 157-8° (Ac deriv., m. 256.5°; di-Bz deriv., m. 210°); the diazo soln. from XII and 1-ClOH₂NH₂ give XI. Redn. of 10 g. XI in 200 mL. boiling AcOH with 20 g. Zn (5 mL. HCl added to complete the reaction) gives 55% 3-(1-naphthyl)-1,2,7,8-dibenzocarbazole, m. 218-19° (a brownish-yellow EtOH complex seps. from abs. EtOH); picrate, maroon, m. 252° (decompn.); the dil. EtOH soln. has an intense blue-green fluorescence in the UV; the pale red concd. H₂SO₄ soln. becomes intense indigo blue with oxidizing agents. An attempt to phenylate (PhN₂)₂ in Me₂CO failed. The mechanism, involving a radical-type reaction, is discussed, although cationoid substitutions by aryl ions cannot be excluded.

ACCESSION NUMBER: 1950:40744 CAPLUS
 DOCUMENT NUMBER: 44:40744
 ORIGINAL REFERENCE NO.: 44:7811b-i, 7812a-i, 7813a-b
 TITLE: Decomposition reactions of aromatic diazo compounds. XII. The reaction between diazo compounds and potassium ferrocyanide
 AUTHOR(S): Ford, M. C.; Waters, William A.; Young, H. T.
 CORPORATE SOURCE: Univ. Oxford, UK
 SOURCE: Journal of the Chemical Society, Abstracts (1950) 833-40
 CODEN: JCSAAZ; ISSN: 0590-9791
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 44:40744

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GI For diagram(s), see printed CA issue.
AB 5,6,7,8-Tetrahydroisquinoline (I) (150 g.) was stirred 5 h. at 140-50° with 225 g. PhNMe₂ and 150 g. powdered NaNH₂, heated another 10 h. without stirring, and the powdered product decomposed with ice, yielding
after Et₂O extraction 85% 1-NH₂ derivative (II) of I, b.p. 45 120-1°, m. 81° (from petr. ether); picrate, pale yellow, m. 269° (from glacial AcOH); HCl salt, m. 228°; HBr salt, m. 182°. To 175 cc. HBr (d. 1.49) at -5°, 40 g. I was added gradually, and the mixture treated dropwise at -10° with 36 cc. Br. The first product is a perbromide, an orange crystalline deposit, which was treated with thorough stirring (at -10°) very gradually with 50 g. dry crystalline powdered NaNO₂, the mixture was cooled after 1 h. to -30°, treated with 160 g. NaOH in 200 cc. H₂O at < 10°, and extracted with Et₂O, giving 45.8 g. 1-Br derivative (III) of I, b.p. 73 127°, m. 48° (from petr. ether), a weak base which could be extracted from its acid solns. with Et₂O (picrate, long, greenish yellow needles, m. 105°), and 5.5 g. 1,4-di-Br derivative (IV) of I, C₉H₉NBr₂, m. 87° (from EtOH) (isolated from the mother liquors of III), which forms no picrate and is soluble in concentrated, but not in dilute acids. (The yields of III were very poor, when, in the diazotization of I, a concentrated aqueous NaNO₂ solution was used.)
When the above conditions of diazotization were otherwise modified (modifications not described) the following byproducts were obtained besides III and IV: 1-amino-4-bromo derivative of I, b.p. 9 152°, m. 140° (from EtOH) (picrate, m. 272°), and 1-hydroxy-1-bromo derivative of I, m. 196° (from aqueous AcOH). From the 1,3-di-HO derivative of I (cf. C.A. 43, 4279a) and PBr₃ was formed the 1,3-di-Br derivative of I, b.p. 12 198°, m. 100°, and from the filtrate the 1-bromo-3-hydroxy derivative of I (isolated as the picrate, m. 131°), together with isquinoline and I. To III (30 g.) in 500 cc. dry Et₂O at -35° under N and 120 cc. 1.2 N BuLi in Et₂O was added 17.5 g. BzH at such a rate that the temperature remained below -25°; when the solution turned pale yellow, the Et₂O solution was washed with H₂O, extracted with 5 N HCl, made alkaline, and reexd. with Et₂O, yielding 18.9 g. 1-PhCH(OH) derivative (V) of I, b.p. 0.5 158-61°, prisms, m. 63° (from petr. ether), forming no picrate but yielding an HCl salt, m. 234° (from EtOH-Et₂O), an H₂SO₄ salt, m. 213° (from EtOH), and a nonhygroscopic C methiodide, m. 201-3° (from Me₂CO). When oxidized at room temperature in dry Me₂CO with KMnO₄, V gave the 1-Bz derivative (VI) of I, oil, isolated as the picrate, m. 160° or the H₂SO₄ salt, m. 166°, and reconverted on catalytic hydrogenation into V. When refluxed with HI (d. 1.7) and red P, V gave 90% 1-PhCH₂ derivative (VII) of I, b.p. 25, 150°, m. 31° (picrate, m. 131.5°; H₂SO₄ salt, m. 214°) methiodide, hygroscopic crystals; methobromide, obtained in 2 nonhygroscopic forms, with 1 H₂O (VIIa), coarse crystals, m. 92° (from Me₂CO), and with 5 H₂O, m. 230°, (I) (from moist MeOH). When 5 g. V in tetrahydro-naphthalene was heated 8-10 h. at 205-10° with 1 g. Pd-C, followed by successive treatment with Et₂O, extraction with HCl, treatment with aqueous NaOH, and reexn. with Et₂O, a 1:1 mixture of VI and VII was obtained, Clemmensen reduction of which gave the Zn double salt of VII, m. 181° (readily converted into VII by aqueous NH₄OH). When heated with PhCH₂Cl, VI gave the N-PhCH₂ adduct of VII, C₂₃H₂₄NCl, m. 98° (from Me₂CO), which when hydrogenated (Pt catalyst)

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contg. iodine when hydrogenated with Pt gave the "pairing body" (XX), m. 202°, giving a wine-red color with Gibbs reagent (indicating substitution on the position para to the OH group); its Me ether, yellow oil, gave a picrate, m. 219°, and its methiodide, m. 268° was unattacked by hot aq. NaOH but underwent the Hofmann degradn. with Ag₂O, yielding an oil which with an excess of MeI gave MeANI. Syntheses similar to those described were also carried out with III and 3,4-(MeO)₂CHCH₂CHO, giving the following derivs. of I: 1-α-hydroxyveratryl (XI), b.p. 0.3 198-200°, m. 90° (from Et₂O-petr. ether) (yielding no cryst. picrate; H₂SO₄ salt, m. 185°; methiodide, m. 163°); 1-veratryl, m. 131° (picrate, pale yellow, m. 105° readily changing in light to an orange modification, m. 155°); 1-veratryl (XXII), b.p. 0.3 195°, m. 66° (from petr. ether) (purified through the picrate, an unstable, pale yellow modification, m. 143°, passing into the stable orange form, m. 174°, identical with XXIII) HCl salt, m. 203°; methiodide, viscous oil; methobromide (3H₂O), m. 55° (1 H₂O, m. 107°); Me₂SO₄ adduct, oil. Demethylation and redn. of XXI yielded the 1-(3,4-dihydroxybenzyl) deriv. of I, whereas Na redn. of XXI yielded the 1-veratryl-1,2,3,4-tetrahydro deriv. of I, b.p. 1 165°, picrate, m. 214-16°. Catalytic hydrogenation of XXI with Cu chromite in abs. EtOH at 200° at 230 atm. gave the N-ethyl-1,2,3,4-tetrahydro deriv. of I, mobile oil, b.p. 13 110° isolated as the picrate, m. 133°. XXII.MeBr in MeOH and NaOH hydrogenated with Pt gave as the main product the N-methyl-1,2,3,4-tetrahydro deriv. (XXIIa) of XXII (picrate, m. 143.5-5°), and as a byproduct in small amts., the N-Me 1,2,3,4,9,10-hexahydro deriv. (XXIIb) of XXII, isolated as the picrate, m. 182°. The methiodide of XXII, m. 185° subjected to the Hofmann degradn. gave the oily desbase, 3,4-(MeO)₂CHCH₂CH:CH:CH₂CH₂CH₂CH₂CH₂CH₂, which on hydrogenation gave the tetrahydro deriv. (XXIII) (picrate, m. 130°). Similarly, Hofmann degradn. of XXIIb.MeI (m. 202°), followed by hydrogenation also gave XXIII. HCl gas passed for 10 h. into 2.9 g. XXII in 40 cc. concd. HCl at 120°, followed by diln. with H₂O, treatment with NH₄OH, and extn. with Et₂O and washing of this ext. with H₂O, aq. NaOH, and H₂O gave 0.2 g. 4-hydroxy-3-methoxy-N-methylmorphinan, identical with di-tetrahydrodesoxycodeine (XXIV), b.p. 0.1 120, hexagons, m. 127-30° (decomp. at 135°). By treatment with the equiv. amt. of d-tartaric acid in dioxane, XXIV gave 1-tetrahydrodesoxycodeine tartrate (contg. 1 mol dioxane), m. 110° (decompn.) (cf. Kondo and Ochiai, C.A. 23, 3709). XXIV could also be formed (but far less effectively and in poor yield) by heating XXII in 50 cc. HBr under N. This treatment, while it results in cyclization, caused removal of both MeO groups, hence partial demethylation was required.
ACCESSION NUMBER: 1950:10119 CAPLUS
DOCUMENT NUMBER: 44:10119
ORIGINAL REFERENCE NO.: 44:1994f-1, 1995a-1, 1996a-1, 1997a-b
TITLE: Total synthesis of tetrahydrodesoxycodeine
AUTHOR(S): Greve, Rudolf; Mondon, Albert; Nolte, Elisabeth
SOURCE: Ann. (1949), 564, 161-98
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 44:10119

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in alk. MeOH took up 1 mol H. After acidification, the mixt. added another 0.9 mol H, yielding the 1,2-dibenzyl-1,2,3,4-tetrahydro deriv. (VII) of I, isolated as the picrate, m. 159°. VIII shaken in neutral MeOH with Pd-C and H lost PhMe and gave the 1-benzyl-1,2,3,4-tetrahydro deriv. (IX) of I, b.p. 3 114°, identified as the picrate, m. 152°. IX was also obtained by reducing V with EtONa in EtOH. The MeI deriv. of IX m. 177-8°. IX (0.05 g.) in 1 cc. H₃PO₄ (d. 1.7) heated 12 h. at 120° gave morphinan (X), identified as the picrate, m. 186° (the picrate reported previously by G. and M., loc. cit., m. 207°; this appears to be another cryst. form; the m.p. of the mixt. lies somewhere between 186° and 207°). VIIa (0.25 g.) in MeOH contg. 1.5 cc. N NaOH was hydrogenated with a Pt catalyst, giving 72% of the 2-Me deriv. (XII) of IX; picrate, m. 133°. XI (0.28 g.) was also formed from the MeI deriv. of VII. The following derivs. of XI were prepd.: HNO₃ salt, m. 161.5°; HBr salt, m. 194.5-5.5°; "MeI deriv.", m. 178° (identical with the deriv. of IX). By analogous methods from III, BuLi, and p-MeOC₆H₄CHO was formed the 1-[p-MeOC₆H₄CH(OH)] deriv. (XII) of I, b.p. 2 195-8°, cryst. in 2 modifications, m. 69° (the more common form), and m. 89-90°; H₂SO₄ salt, m. 199° (decompn.); MeI adduct, m. 146°; XII formed no picrate. Oxidn. of XII gave the 1-anisoyl deriv. of I, m. 67.5° (from petr. ether), purified through its picrate and redn. of XII with HI gave the 1-[p-HOC₆H₄CH₂] deriv. (XIII) of I, m. 168° (from C₆H₆). When XII was treated with Zn in HBr-AcOH or with Pd-C in tetrahydronaphthalene, or when XIII was treated with CH₂N₂, the 1-anisyl deriv. of I, m. 39°, was formed (best purified through its picrate, m. 147° (another form with 2 mol H₂O m. 154°)); MeI deriv. (XIV), m. 167.5°; CH₃Cl deriv., noncryst. Catalytic hydrogenation of XIV (or the Cl analog) gave the 2-methyl-1-anisyl-1,2,3,4-tetrahydro deriv. (XV) of I, oil; HCl salt, m. 151°; picrate (XVI), m. 174°. Another picrate, C₂₄H₃₀N₄O₈, m. 205°, isolated in varying amts. from the mother liquors of XVI, contains 1 mol H more than does XVI. By heating 0.25 g. XV 6 h. with 2.5 cc. (48%) HBr at 135° there was formed the 3-hydroxy-N-Me deriv. (XVII) of X, m. 252° (cf. Schneider and Grussner, C.A. 43, 6635c); HBr salt, m. 200°; 3-Me ether, m. 83° (picrate, m. 168°). By appropriate reactions, the following derivs. of XVII were formed. 2-Br, m. 236-43° (decompn.) (picrate, m. 145°); 2-bromo-O-Me, m. 152.5° (subliming in vacuo at 70°) (picrate, m. 238-9°); 2,4-di-Br, decomp. 223-4° (purified through its HBr salt, m. 215-17°); 2,4-dibromo-O-Me, oil (picrate m. 190°); 2-iodo, m. 196° (HCl salt, m. 223-4°; HBr salt, m. 223°; picrate, m. 162-68°), readily reconverted into XVII when heated with aq. Ba(OH)₂ and small amts. of Cu-bronze; 2-iodo-O-Me, m. 178.5° (picrate, m. 238°), as well as 2 other unexplored picrates, deep yellow, m. 176°, and ruby-red, m. 187°; 2-hydroxy-O-Me, C₁₈H₂₅N₂O₂, m. 189° (from dry Me₂CO), coupling with O₂NCH₂CH₂NO₂ to give a reddish-brown dye; 2-MeO-O-Me, oil, giving 2 forms of picrate, m. 211° and 185° and a methiodide, m. 237°; 2-HO, m. 139° (best isolated through its HBr salt, m. 246°). Starting with isovanillin, II, and BuLi, the following derivs. of I were prepd. by methods analogous to those described: 1-[4,3-MeO(PhCH₂)C₆H₃CH(OH)] (XVIII), m. 85-7°, isolated through its HCl salt, m. 177.5°; 1-[4,3-MeO(HO)C₆H₄CH(OH)] (XIX), m. 179° (from alic.-Et₂O), giving a blue color with Gibbs reagent; isovanillyl, m. 205° (whose mono-Me ether gives a picrate, m. 174° (XXVIII)); 1-[3,4-AcO(HO)C₆H₃CH₂] (XXIX), m. 74° (picrate, m. 118°). The hygroscopic, cryst. XIX.MeI in MeOH-NaOH

L20 ANSWER 228 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
AB The 85-15 copolymers of methyl methacrylate-maleic anhydride react with alic., amines, and metallic salts to produce clear products which are solvent-, heat-, and abrasion-resistant; Esterification of 85-15 copolymer and primary and secondary aromatic and aliphatic alics. react to produce half-esters at 70° in the presence of 0.5% Na₂CO₃ catalyst. The products are softer than the original. Resistance to solvents and heat is increased when ethylene glycol, glycerol, and diethylene glycol are used. Amiation, solns. of aniline, o- and p-chloroaniline, p-bromoaniline, o- and p-aminophenol, 2-amino-4-nitrophenol, p-aminodimethylaniline, sulfanilic acid, p-aminobenzoic acid, p-aminobenzenesulfonamide, p-aminobenzophenone, p-aminobiphenyl, 2-amino-p-cymene, 1-aminocanthraquinone, α-naphthylamine, 2,4-diaminodiphenylamine, dibutylamine, and octadecylamine in aqueous or anhydrous MeOH produce materials which have increased shear hardness, equivalent mar resistance, but sometimes colored or crazed surfaces compared with the original copolymer. Metal salts, with cations of H, NH₄⁺, Ti⁴⁺, Ag, Cu⁺, K, Zn, Mg, Mn²⁺, Ca, Cd, Ni²⁺, Ba, Sr²⁺, Al, Cr³⁺, Hg²⁺, Fe³⁺, Co²⁺, Cu (NH₃)₄²⁺, Sr²⁺, UO₂²⁺, show generally improved shear hardness, scratch resistance and greater percentage transmission than the original copolymers.
ACCESSION NUMBER: 1949:45509 CAPLUS
DOCUMENT NUMBER: 43:45509
ORIGINAL REFERENCE NO.: 43:8202a-e
TITLE: Surface reactions of copolymers. Methyl methacrylate and maleic anhydride
AUTHOR(S): Seymour, Raymond B.; Brannan, Ira, Jr.; Hayward, F. V.
SOURCE: Journal of Industrial and Engineering Chemistry (Washington, D. C.) (1949), 41, 1482-4
CODEN: JIECAD; ISSN: 0095-9014
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L20 ANSWER 229 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB To 8.4 g. powdered PC15 in 100 mL. alc.-free, dry CHCl₃ was added, gradually, 8.4 g. 9-(2-diamylamino-1-hydroxyethyl)-1,2,3,4-tetrahydrophenanthrene-HCl (I), the mixture shaken cold until the solid dissolved, left overnight at room temperature, concentrated in vacuo, the residue extracted with 100 mL. absolute Et₂O to remove F halides, the mixture cooled in a refrigerator until it had crystallized, the Et₂O solution decanted, the residue decomposed by shaking with ice, Et₂O, and small amts. of Na₂CO₃, the Et₂O layer separated, washed, dried with Drierite, concentrated in vacuo, an equivalent amount of HCl in MeOH added, and the mixture concentrated in vacuo to a sirup which could be crystallized with Et₂O and isopentane, giving 8.0 g. 9-(1-chloro-2-diamylaminoethyl)-1,2,3,4-tetrahydrophenanthrene-HCl (SN 8845) (III). This, after 3 recrystns. from MeOH by addition of Et₂O and isopentane, sintered at 82°, m. 89-91°; after several weeks in a desiccator, it m. 95°. A mixture of 0.3 g. catalyst (PdOH, Zn(OH)₂, and Cu(OH)₂ on CaCO₃), 2.7 g. CaCO₃, and 75 mL. MeOH was shaken with H to reduce the catalyst, then 2.2 g. II added, the mixture shaken with H until no more was absorbed (95 mL. H in 45 min.; theory, 124 mL.), filtered, concentrated in vacuo, the residue extracted with Et₂O plus a small amount of aqueous NaOH, and the Et₂O solution washed, dried, concentrated, and subjected to evaporative distillation, giving 1.5 g. yellow oil between 100° and 150° at 0.1 mm. This was dissolved in Et₂O and mixed with 1.5 g. picric acid in absolute EtOH; concentration of the solution gave 1.5 g. crystalline 9-(2-diamylaminoethyl)-1,2,3,4-tetrahydrophenanthrene picrate (III) (SN 11,580), m. 110-11° after 3 recrystns. from absolute EtOH. The picrate of I sintered at 142°, m. 150-1°, after 4 recrystns. from absolute EtOH. The 9-(2-diamylaminoethyl)-1,2,3,4-tetrahydrophenanthrene (not prepared pure as such) could also be characterized as its perchlorate, 1-maleate, and sulfate. The latter, prepared from III in 100% yield, m. 124-7°. II showed about the same antimalarial activity as I, but the sulfate prepared from III was inactive.

ACCESSION NUMBER: 1949:41406 CAPLUS
 DOCUMENT NUMBER: 43:41406
 ORIGINAL REFERENCE NO.: 43:7469b-1,7470a-d
 TITLE: Functional derivatives of the antimalarial 9-(2-diamylamino-1-hydroxyethyl)-1,2,3,4-tetrahydrophenanthrene (SN 1796)
 AUTHOR(S): Richtmeyer, Nelson K.
 SOURCE: Journal of Organic Chemistry (1949), 14, 334-6
 CODEN: JOCEAH; ISSN: 0022-3263
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 231 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB cf. C.A. 41, 6884c. The present work is a continuation of the earlier studies on antitubercular compds., but no pharmacol. data are given. 2,5-C1(O2N)C6H3CO₂H (I) (85 g.) in 800 mL. 95% EtOH, treated slowly with 400 mL. H₂O and then with 103 g. p-ICG4SH (II) and 73.2 g. NaHCO₃, and the mixture refluxed 22 hrs., gives 37 g. of 5-nitro-2-(p-iodophenylmercapto)benzoic acid (III), bright yellow, m. 244-7°, and 40 g. (p-ICG4)2S₂. I (16 g.), 1.7 g. p-BrC₆H₄SH, 1.2 g. KOH, and a trace of Cu in 90 mL. EtOH, refluxed 22 hrs. in a N atmosphere, give 36% of the p-Br analog (IV) of III, yellow, m. 224-6°. 2,5-C1(O2N)C6H3CHO (74.2 g.), 98.4 g. II, 35 g. NaHCO₃, 850 mL. 95% EtOH, and 450 mL. H₂O, refluxed 2 hrs., give 84% 5-nitro-2-(p-iodophenylmercapto)benzaldehyde (V), yellow, m. 131-3°, p-Br analog (VI), yellow, m. 123-5°, 98%. III (1.6 g.) and 2.8 mL. 30% H₂O₂ in 35 mL. AcOH, refluxed 3 hrs., give 81% 5-nitro-2-(p-iodophenylsulfonyl)benzoic acid, m. 242-6° (decomposition); p-Br analog m. 190.5-2°, 90%. V (10 g.), in 100 mL. cold concentrated H₂SO₄ poured onto 150 g. ice, with crystallization of the product from Me₂CO, gives 83% 2-iodo-8-nitrothioxanthone (VII), yellow, m. 155.5-7.5°, and 91% 2-iodo-8-nitrothioxanthone (VIII), yellow, m. 300-3° (decomposition). III (20 g.) in 180 mL. concentrated H₂SO₄, kept 90 min. at 100°, gives 84% VIII. VI gives 67% of the 2-Br analog (IX) of VII, yellow, m. 162-4.5°, and 92% of the Br analog (X) of VIII, bright yellow, m. 282-5° (decomposition); IV gives 95% of X. The solution prepared by passing dry HCl into 4.1 g. SnCl₂.2H₂O in 22 mL. AcOH, treated with 1.6 g. IX in 6 mL. AcOH at 80-5°, heated 2 hrs. at 80-5°, and the Sn complex decomposed with cold 10% NaOH, gives 96% 2-bromo-8-aminothioxanthone (XI), brownish yellow, m. 183-4°. VII (1 g.) and 3 mL. 30% H₂O₂ in 30 mL. AcOH, refluxed 3 hrs. and 8 mL. 30% H₂O₂ added during the next 4 hrs., give 92% 2-iodo-8-nitrothioxanthone 5-dioxide (XII), m. 229-31°. IX (1.8 g.) and 1.5 mL. 30% H₂O₂ in 30 mL. AcOH, refluxed 2 hrs. and the product crystallized from AcOH, gives 28% unreacted IX, 15% 2-bromo-8-nitrothioxanthone 5-dioxide (XIII), pale yellow, m. 273-5°, and 41% 2-bromo-8-nitrothioxanthone 5-dioxide (XIV), pale yellow, m. 205-7°. 2-iodo-8-nitrothioxanthone 5-dioxide (XV), m. 303-6° (decomposition), was prepared in 3 ways. XII (0.8 g.), 0.5 g. CrO₃, 20 mL. AcOH, and a few drops concentrated H₂SO₄, refluxed 90 min., give 0.7 g. XV. VIII is not attacked by refluxing for prolonged periods with a large excess of 30% H₂O₂ in AcOH (VII and VIII are catalysts for the decomposition of H₂O₂ but IX is not as effective). VIII (1 g.), CrO₃, and AcOH, refluxed 2 hrs., give 92% XV. A mixture of VII and VIII with CrO₃ gives 95% XV (based on V). The mixture of IX and X from the ring closure of 43.2 g. VI with 430 mL. H₂SO₄, oxidized in 950 mL. AcOH by refluxing 4 hrs. with 130 mL. 100% H₂O₂, gives 79% XIII; it results also by oxidation of X with excess 30% H₂O₂ in boiling AcOH and from the mixture of IX and X with excess CrO₃ in AcOH. XV (116 g.), reduced with 241 g. SnCl₂.2H₂O, yields 87% 2-iodo-8-aminothioxanthone 5-dioxide (XVI), bright yellow, m. 301-4° (decomposition); 0.5 g. XVI, 5 mL. 12 N NH₄OH, and a trace of Cu, heated 22 hrs. at 220°, give 0.3 g. 2,8-diaminothioxanthone 5-dioxide (Part III); XVI with Zn and aqueous-alc. alkali gives a deep violet color which is discharged on exposure to air. 2-Br analog (XVII) of XVI, yellow-orange, m. 310-11°, quant. yield. XVI (5 g.), 15 g. 2-aminopyrimidine, 2 g. powdered anhydrous K₂CO₃, and a trace of Cu, heated 80 min. at 195-205°, and the product triturated with hot H₂O, and extracted with cold 5 N HNO₃, give 90% 2-amino-8-(2-pyrimidinylamino)-thioxanthone 5-dioxide, bright yellow, m. 335-8° (decomposition); di-Ac derivative, yellow-orange, m. 318-21° (decomposition), 66%. XVI (2 g.), 6 g.

L20 ANSWER 230 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 GI For diagram(s), see printed CA issue.
 AB cf. 2 preceding abstrs. In the du Pont labs. 2-furaldehyde (I) has been utilized as a raw material for further syntheses. Thus, a mixture of I and steam at 400° is passed over a catalyst consisting of a mixed chromite of Zn and either Mn or Fe to give furan (II) and CO. II is hydrogenated to 0.CH₂.CH₂.CH₂.CH₂ (III), f.p. -107 to -108°. III is a versatile starting material leading to such products as BuOH, 0.CH₂.CH₂.CH₂.CO (with O at 120° and 100-200 lb. over a Co catalyst) or (HO₂CCH₂)₂, CH₂.CHCH₂.CH₂ (by passage with steam over a phosphate catalyst at 270°), 0.CH₂.CH₂.CHCl.CHCl (chlorination at moderate temps. under the influence of light or other activating agent), HO(CH₂)₄Cl, Cl(CH₂)₄Cl (IV) (with aqueous HCl under 15-20 atmospheric and 180°), (AcO-CH₂CH₂)₂ and polymerization to [O(CH₂)₄O(CH₂)₄]_n (SOC1₂ and FeCl₃ as catalysts) and copolymers with 0.CH₂.CH₂ and 0.CH₂.CHMe. IV and NaCN give Cl(CH₂)₄CN which with Na₂S gives [NC(CH₂)₄]₂S (V). V and the corresponding sulfone can be converted into the acid, diamide, and esters resembling the sebacates in many properties. IV and NaCN also give NC(CH₂)₂CN. The hydrogenation of I to 0.CH₂.CH₂.CH₂.CHCH₂OH (VI) and catalytic dehydration of VI gives 0.CH₂.CH₂.CH₂.CH₂.CH₂ (VII). VII functions as a source of 0.CH₂.CH₂.CH₂.CH₂.CH₂, HO(CH₂)₅OH (VIII) HO(CH₂)₅Cl, Cl(CH₂)₅Cl, Cl(CH₂)₅CN, NC(CH₂)₅CN, H₂N(CH₂)₅NH₂, HO₂C(CH₂)₅CO₂H, [NC(CH₂)₅]₂S, and [NC(CH₂)₅]₂SO₂ and their corresponding acids, diamines, and esters, 0.CH₂.CH₂.CH₂.CH₂.CO (VII) over Co catalysts in air with H₂O or by passage of VIII over Cu at elevated temps.), 0.CHCl.CHCl.CH₂.CH₂.CH₂, 0.CH:OCl.CH₂.CH₂.CH₂, 0.CH(OH).CHCl.CH₂.CH₂.CH₂, and 0.CHCl.CH₂.CH₂.CH₂.CH₂. VII and COCl₂ give unstable derivative, which upon heating gives a fair yield of 2,3-dihydro-1,4-pyran-5-carbonyl chloride, which can serve as the starting point for a variety of interesting new compounds.

ACCESSION NUMBER: 1948:21347 CAPLUS
 DOCUMENT NUMBER: 42:21347
 ORIGINAL REFERENCE NO.: 42:4565f-1,4566a-b
 TITLE: Chemical intermediates from furfural
 AUTHOR(S): Cass, Oliver W.
 CORPORATE SOURCE: E. I. du Pont de Nemours & Co., Inc., Niagara Falls, NY
 SOURCE: Journal of Industrial and Engineering Chemistry (Washington, D. C.) (1948), 40, 216-19
 CODEN: JIECAD; ISSN: 0095-9014
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 231 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN (Continued)
 2-aminopyridine (XVIII), 0.8 g. K₂CO₃, and a trace of Cu, heated 90 min. at 190-5°, the tarry mass extd. with warm H₂O and then with 6 N HCl, and the ext. treated with excess NH₄OH, give 0.4 g. of an orange compd., C₁₈H₁₃O₃N₃S, m. 235-40° (decompn.); refluxed with aq.-alc. alkali, it evolves NH₃; it is possible that the XVIII may have reacted, in part, as 2-aminopyridone. XVI (1 g.) and 3 g. 2-bromopyridine, heated 8 hrs. at 155-60°, give 83% 2-iodo-8-(2-pyridylamino)thioxanthone 5-dioxide (XIX), orange, m. 294-5°, the 2-Br analog (XX), orange, m. 297-8°, 92%, 2.6 g. XX, 12 mL. 12 N NH₄OH, and a trace of Cu, heated 20 hrs. at 210-20°, give 68% 2-amino-8-(2-pyridylamino)-thioxanthone 5-dioxide, yellow-orange, m. 280-2° (decompn.); XIX gives the same compd. XVI (1 g.) and 2.6 g. 2-chloroquinoline, heated 15.5 hrs. at 150-4°, 54 hrs. at 155-65°, 18.5 hrs. at 160-70°, and to 185° during the last hr., give 79% crude 2-iodo-8-(2-quinolylamino)-thioxanthone 5-dioxide, m. 320-1° (decompn.); with concd. NH₄OH at 205° (15 hrs.), this yields 3% of the 2-amino analog. The thioxanthone dioxides gives characteristic colors (green to violet) with Zn dust in aq.-alc. alkali; the thioxanthone dioxides give similar colors directly on treatment with cold dil. aq.-alc. alkali; these colors disappear quickly on exposure to air.

ACCESSION NUMBER: 1948:13729 CAPLUS
 DOCUMENT NUMBER: 42:13729
 ORIGINAL REFERENCE NO.: 42:2971e-1,2972a-g
 TITLE: Sulfone series. IV. Certain heterocyclic derivatives of 2,8-diaminothioxanthone 5-dioxide
 AUTHOR(S): Amstutz, E. D.; Fehnel, E. A.; Runzberger, I. M.
 CORPORATE SOURCE: Lehigh Univ., Bethlehem, PA
 SOURCE: Journal of the American Chemical Society (1948), 70, 133-8
 CODEN: JACSAT; ISSN: 0002-7863
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 42:13729

L20 ANSWER 232 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Lazier and Adkins (C.A. 18, 1992) showed that PhNH₂ (I) could be alkylated with the requisite Al alkoxide by heating at 250-350°. It is possible to alkylate salts or metal complexes of I at considerably lower temps. than that for I itself. By the use of 4 times the quantity of Al(OEt)₃ required for complete diethylation, the following quantities of I (in %) were found in the product: 1.HCl, 184°, 5 hrs., 91.8; 1.HBr, 184°, 5 hrs., 46.7; 1.HI, 184°, 5 hrs., 2.4 (18.6% tertiary base); 1.HBr, 198°, 5.5 hrs., 30; 1.H₃PO₄, 198°, 5.5 hrs., 91.3; 1.H₂SO₄, 240°, 5 hrs., 30. On heating 5 hrs. at 240°, the following quantities of I (in %) were present in the product: 21.CdSO₄ 69.8, 21.ZnSO₄ 80.3, 21.ZnCl₂ 56.2, 21.ZnBr₂ 41.8, 21.NiCl₂ 90.3. The addition of Na halides to reaction mixts. containing 1.HX generally increased the rate of alkylation. On heating 5 hrs. at 184°, the addition of 1 and 2 moles NaCl to 1.HCl gives products with 44.1 and 27.4% I; 1 and 1.5 moles NaBr, added to 1.HBr, give products containing 43.6 and 34.3% I. The reaction of Al(OBu)₃ with 1.HCl containing 1 mole of Na halide at 198° gave the following yields (in %) of primary, secondary, and tertiary bases: NaCl, 16 hrs., 65.1, 34.9, -; NaBr, 10 hrs., 43.3, 48.7, 8.0; NaI, 10 hrs., 3.3, 64.0, 32.7. The methods of analysis of the bases are given. The vapor-phase methylation of PhNHMe by MeOH in the presence of Al₂O₃ was studied in an exploratory manner and the data given are only roughly quant. Results of the addition of iodine, MeI, and 1.MeI show that these compds. have a strongly accelerating effect on the reaction and that the effect is retained by the catalyst for some time. When 1.MeCl is added, there is no marked increase in N-alkylation but a certain degree of nuclear alkylation occurs. The data show that a lag must be expected when a promoter is introduced with the reaction mixture, the promoted catalyst not showing its full activity for some time. The effect of the addition of NaCl, NaBr, NaI, NiCl₂, CuCl₂, CoCl₂, MnCl₂, ZnCl₂, CdCl₂, FeCl₃, and SnCl₂ has been studied. With the exception of NaCl, all the salts increased the activity of the catalyst at 250°. Raising the temperature from 250 to 290° increased the rate of alkylation with pure Al₂O₃ and when Na halides, MnCl₂, or ZnCl₂ was present; CoCl₂ and CdCl₂ definitely depressed the rate of alkylation at the higher temperature. Although with pure Al₂O₃ there is no significant quantity of nuclear alkylation even at 290°, most of the added salts promote nuclear alkylation at that temperature and sometimes at 250°, also.

ACCESSION NUMBER: 1948:685 CAPLUS
 DOCUMENT NUMBER: 42:685
 ORIGINAL REFERENCE NO.: 42:136f-1,137a-c
 TITLE: Experimental study of the alkylation of aromatic amines with (1) aluminum alkoxides and (2) alumina with alcohols
 AUTHOR(S): Campbell Earl, J.; Hills, Noel G.
 CORPORATE SOURCE: Univ. of Sydney, Australia
 SOURCE: Journal of the Chemical Society, Abstracts (1947) 973-6
 CODEN: JCSAAZ; ISSN: 0590-9791
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 233 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 AB giving 85% 3,4,5-triaminoveratrole (XII), m. 146-50°. Instead of isolating XII, it can be converted into 8-amino-6,7-dimethylquinoxaline (XIII), m. 106.5-7.5°. Condensation of 12 g. XIII in 75 cc. dry CSH₃N with p-AcNHCH₂SO₂Cl gives 98% 8-(N4-acetyl-sulfanilamido)-6,7-dimethylquinoxaline (XIV), m. 238.5-9° (decomp.). Sapon. of XIV by refluxing it with 3 N HCl gives 8-sulfanilamido-6,7-dimethylquinoxaline (SN 9162), pale yellow prisms, m. 217.5-18.5° (decomp.). Attempts to substitute the H in the NH₂ group in XIII by R₂N(CH₂)₃ met with great difficulties. A mixt. of 7.5 g. XIII in 75 cc. PhOH and 9.4 g. Et₂NCH₂CH₂Cl (XV) is heated 7 hrs. at 100°. The mixt. is then acidified and steam-distd. to remove the PhOH. The distn. residue is made alk., extd. with C₆H₆, and the dried C₆H₆ ext. distd., giving 8-(2-diethylaminoethylamino)-6,7-dimethylquinoxaline (SN 12,366), viscous orange oil, b_{0.5} 175°. Attempts to use Et₂N(CH₂)₃CH₂Br in lieu of XV failed. Diazotization of 2.05 g. XIII, making the soln. alk., and decomp. the diazonium salt by heating give VII, m. 238-9°. In an attempt to replace the NH₂ group in XIII by iodine by a Sandmeyer reaction, an iodo deriv. (XVI), nacreous pale yellow leaves, decomp. about 210° without melting, is formed. When XVI is treated with Et₂N(CH₂)₃CH(NH₂)Me in BuOH, VII is obtained. When the diazonium salt of XIII is heated 1 hr. with NaOAc and Cu powder at 100°, 11.9% VII, m. 238-9°, is formed. From the mother liquor a compd., sandy pale orange crystals, m. 163-4°, is isolated; when heated with 2 N NaCO₃ it also gives VII. None of the compds. shows antimalarial activity.

ACCESSION NUMBER: 1947:37353 CAPLUS
 DOCUMENT NUMBER: 41:37353
 ORIGINAL REFERENCE NO.: 41:7400d-1,7401a-f
 TITLE: Experiments in the veratrole and quinoxaline groups
 AUTHOR(S): Ehrlich, Jacob; Bogert, Marston T.
 CORPORATE SOURCE: Columbia Univ.
 SOURCE: Journal of Organic Chemistry (1947), 12, 522-34
 CODEN: JOCEAH; ISSN: 0022-3263
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 41:37353

L20 ANSWER 233 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Some veratrole and quinoxaline derivs. are prepared for the study of their pharmacol. activity. To 192 cc. concentrated HNO₃, 69 g. veratrole is added dropwise at 0-3° over a period of 80 min. After stirring 5 min., 105 cc. concentrated H₂SO₄ is added over a period of 1 hr. at 3-5°. The mixture is warmed within 20 min. to 54-5°, kept there for 10 min., and then at 58-60° 10 min. The partially crystalline mixture is poured into 400 cc. ice-H₂O and diluted with 2500 cc. H₂O, giving 94-6% 4,5-dinitroveratrole (I), fine pale yellow needles, m. 130-1°. I (5 g.) refluxed 11 hrs. with 40% HBr gives 1.15 g. 4,5-dinitroquinoxalol (II), hairlike, pale yellow needles, m. 172-3°. II is isolated via its scarlet Na salt; its NH₄ salt, orange crystals, is obtained by precipitation with 15 N NH₄OH. From the alkaline mother liquor of II, 1.12 g. 4,5-dinitropyrocatechol (III), sandy canary-yellow crystals, m. 166.5-7.5° (decomposition), is isolated (di-Na salt (IV), dark steel-blue crystals, explodes violently on heating). When a suspension of dry IV in PhMe is refluxed 16 hrs. with Me₂SO₄, I and II are formed. Reduction of III with Fe, Zn, or in alc. solution in the presence of Adams catalyst gives impure products. When 2 g. III in 20 cc. H₂O is reduced with 14.5 g. 90% Na₂S₂O₄ in 80 cc. H₂O at 0-5°, the mixture gradually warmed to 65-70°, chilled, decomposed with 10 cc. AcOH, and 3 g. (CHO)₂.2NaHSO₃ (V) is added, the mixture stirred 10 min. at 28-30°, then heated 1 hr. at 95-100°, 81% 6,7-dihydroxyquinoxaline (VI), subliming at 255°/1 mm., yellow crystals, darkening at 260° and charring without melting, is obtained. VI is soluble in dilute Na₂CO₃ and NH₄OH; HCl salt, violet crystals, hydrolyzes in H₂O to give VI. Reduction of II with Fe-HCl in alc. and condensation of the 4,5-diaminoquinoxalol with V give 6-methoxy-7-hydroxyquinoxaline (VII), silvery nacreous plates, m. 238-9°. Neither the Cu salt, nor the canary-yellow Ag salt shows any evidence of chelate formation. Bz derivative of VII, pale tan nacreous leaves, m. 136.5-7.5°. Reduction of I with Fe-HCl in alc. and condensation of the 4,5-diaminoveratrole with V give 6,7-dimethylquinoxaline (VIII), long needles, m. 150-1°. When VIII is refluxed with 40% HBr an insol. brown resin is formed and with 30% HBr VIII is recovered unchanged. Slow addition of 11.4 g. VIII to 120 cc. 95.5% H₂SO₄ at 30 cc. concentrated HNO₃ over a period of 1 hr. at 23-5°, keeping the mixture 40 hrs., and pouring it into 1380 cc. ice-H₂O give a precipitate which is dissolved while still moist in 22 cc. 2 N Na₂CO₃. The insol. 8-nitro-6,7-dimethylquinoxaline (IX) (39.7%) is filtered off and recrystd. from EtOH, hairlike needles, m. 122-4°. From the blood-red filtrate, 22.2% 6-methoxy-7-hydroxy-8-nitroquinoxaline, pale yellow crystals, darkening at 235° and charring without melting, is precipitated on acidification; Na salt, fine bright orange needles, is not methylated when treated with MeI, Me₂SO₄, or p-MeC₆H₄SO₃Na. Reduction of IX with Fe and AcOH at 93-5° gives 8-amino-6,7-dimethylquinoxaline (X), m. 107.5-8.5°. Nitration of veratrole with a mixture of concentrated HNO₃ and concentrated H₂SO₄ gives 3,4,5-trinitroveratrole (XI), m. 144.5-5.5°. To a mixture of 33.5 g. steel card teeth (Number 33 V & M gage wire) in 190 cc. EtOH and 105 cc. concentrated HCl, 1.4 cc. 10 N SnCl₂ is added, followed by 9.1 g. XI. The mixture is gradually heated and refluxed 1 hr. Then 570 cc. is distilled off. The cooled mixture is filtered, the residue washed, 8 g. Na₂SO₃ is added, and the Fe precipitated by addition of Na₂CO₃. The filtrate is bleached with a little Na₂S₂O₄ and extracted with CHCl₃.

L20 ANSWER 234 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB cf. C.A. 40, 1449.3. Several reactions of C₂H₂ under pressure with various catalysts provide compds. useful in preparing plastics. With hydroxy compds. and alkali catalysts vinyl ethers, ROCH₂CH₂, are prepared where R is aliphatic (I), cycloaliphatic (II), aromatic (III), oxygen-containing heterocyclic (IV), and diethylamino-substituted aliphatic. II polymerize to clear, colorless resins; III to hard, dark-colored resins; IV to lacquer-compatible resins. Diethylaminoethyl vinyl ether (V) can be polymerized only in liquid SO₂. Copolymerization of I with acrylic esters and vinyl chloride takes place, while V copolymerizes with acrylic esters in MeOH-H₂O emulsions. III with HCN and pyridine catalyst form α-alkoxypropionitriles in quant. yields. III with NH₃ and Cu-containing catalysts form 2-methyl-5-ethylpyridine. Phenols and C₂H₂ with Zn and Cd salts or amines as catalysts form polyhydroxystyrenes. p-Butylphenol, triphenylolpropane, and hydroxyphenylmethane form resins useful as adhesives and in the preparation of thermosetting resins with hexamethylenetetramine. Aldehydes and C₂H₂ with Ag or Cu acetylides as catalysts form alkynols and alkynediols. CH₂O yields propargyl alcohol and 2-butyne-1,4-diol (VI), from which are prepared 1,4-butanediol, tetrahydrofuran, butadiene, and 68 derived compds. all of which are tabulated in a flow sheet. With CH₃CHO 16 products, also presented in flow sheet form, are derived from 3-butyne-2-ol and 3-hexyne-2,5-diol. The preparation of 1,2,4-butanetriol from VI is detailed in a flow sheet. Tables show the preparation of 8 dicarboxylic acids from di- and tetrahydrofuran and butyrolactone, available from the CH₂O reaction, and from cyclooctatetraene (VII). Cyclopolyolefins prepared by polymerization of C₂H₂ over Ni catalysts are VII, in 90-5% yields, cyclodecapentane, and cyclododecahexane. Suberic acid is prepared from VII.

ACCESSION NUMBER: 1946:14090 CAPLUS
 DOCUMENT NUMBER: 40:14090
 ORIGINAL REFERENCE NO.: 40:2685f-i,2686a
 TITLE: Acetylene as the basis of new plastics
 AUTHOR(S): Reppe, W.
 CORPORATE SOURCE: I. G. Farbenindustrie, Ludwigshafen
 SOURCE: Modern Plastics (1946), 23(No. 6), 169-76, 218, 220
 CODEN: MOPLAY; ISSN: 0026-8275
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 235 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Cyclic O compds. such as cyclohexanol and cyclohexanone are produced by heating to 80-400° (in the absence of added free H) alicyclic monoamino compds., such as cyclohexylamine (I), dicyclohexylamine (II), and phenylcyclohexylamine in the presence of an excess of H₂O or lower aliphatic alc. and in the presence of a hydrogenation catalyst. Thus over a catalyst containing Ni 100, Cr₂O₃ 2, ZnO 1.5, and BaO 2 parts deposited on pumice stone and reduced in H at 300-25° are passed at 180° the vapors of 1 part I and 3 parts H₂O at the rate of 7 g. I per hr. for 1 l. catalyst. Condensation of the vapors and distillation yields 80% cyclohexanol containing a little cyclohexanone and 20% II. The II is added to I in the next charge, and the conversion then amounts to 93-54. 1 100, H₂O 100 parts, and the same catalyst 10 parts are boiled 48 hrs. under reflux to yield 84 parts cyclohexanol and 15 parts amino compds. which can be converted to cyclohexanol by the same process. Other examples of heating I with H₂O, MeOH, or BuOH in the presence of reduced WS₂ and reduced Cu-Zn-Ba catalysts are given under a number of pressure conditions. The variations of the process are discussed.

ACCESSION NUMBER: 1946:8497 CAPLUS
 DOCUMENT NUMBER: 40:8497
 ORIGINAL REFERENCE NO.: 40:1541c-f
 TITLE: Cyclic oxygen compounds
 INVENTOR(S): Schmidt, Willi; Seydel, Karl
 PATENT ASSIGNEE(S): Alien Property Custodian
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2387617		19451023	US	

L20 ANSWER 237 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 G1 For diagram(s), see printed Ca Issue.
 AB PhCH(CH₂NO₂)CH₂Bz (150 g.) and 430 g. HCO₂NH₄ (I), heated 0.25 h. at 180° and 0.25 h. at 190° (evolution of NH₃, followed at 180° by HCO₂H), the product triturated with MeOH and the residue crystallized from PhNO₂, give 40.7 g. of 2,2',4,4'-tetraphenylazadihydropyromethine ((II), R = R' = Ph), dark Cu-colored needles, m. 287-8°; II sublimes without decomposition at 15 mm. and with some decomposition at atmospheric pressure; the very pale blue-violet EtOH solution becomes a deep greenish blue on addition of a drop of mineral acid, from which II is precipitated by alkali; the cold concentrated H₂SO₄ solution gives II if diluted immediately, but if kept for 2-3 h. and then diluted, a brilliant green powder results. The MeOH extract gives 0.62 g. of 2,4-diphenylpyrrole (III), m. 180°. II and Cu(OAc)₂ in BuOH, refluxed 0.5 h., give the Cu salt of II, C₂₄H₁₄N₂O₂Cu, Cu-brown prisms; the Co, Ni and Zn complexes were prepared also. The use of NH₄OH in place of I gives a small amount of II; alc. NH₃ gives a reddish tar but no II; low yields of II result with CO(NH₂)₂ or CS(NH₂)₂; no color resulted with AcNH₂, PrCONH₂ or hexamethylenetetramine (IV); lauramide gave a dull red color; the formates of MeNH₂, PhCH₂NH₂ or H₂N(CH₂)₆NH₂ give low yields of II but that of PhNH₂ gave no color. II and 65% HI, refluxed 0.75 h., give III; neutral oxidation of II with KMnO₄ gave BzH and BzOH; acid oxidation yielded only BzOH; the reaction with other reagents is discussed briefly. 3-Nitrochalcone (25.3 g.) and 9.1 g. MeNO₂ in 200 cc. MeOH at 35°, treated with MeONa (3.45 g. Na in 100 cc. MeOH) and refluxed 10 min., give γ-nitro-β-(m-nitrophenyl)butyrophene, m. 74-7°, on heating with I for 0.25 h. at 180°, there results the 4,4'-bis(m-nitrophenyl) analog of II (R = m-O₂NC₆H₄), needles with bright green reflex, m. 330°. 3-Hydroxychalcone and MeNO₂ give γ-nitro-β-(m-hydroxyphenyl)butyrophene, pale yellow-brown, m. 96-8°, heated with 5 parts of I at 160° this yields the 4,4'-bis(m-hydroxyphenyl) analog of II, needles with violet reflex, m. 304-6°. 4-Dimethylaminochalcone (10 g.) and 3.6 g. MeNO₂ yield 0.5 g. of γ-nitro-β-(p-dimethylaminophenyl)butyrophene, pale yellow, m. 114-15° (oxime, m. 121-3°); with 5 parts of I this yields the 4,4'-bis(p-dimethylaminophenyl) analog of II, needles with bright Cu reflex, m. 276-8°. MeI in PhNO₂ for 24 h. gives a dimethiodide, soluble in H₂O and EtOH with a blue color; it is a wool dye. CH₂O₂C₆H₃CH₂(CH₂NO₂)CH₂Bz (50 g.) and 75 g. I give the 4,4'-bis(3,4-methylenedioxyphenyl) analog of II, Cu-brown needles, m. 258-9°. p-MeOC₆H₄COCH:CHPh and MeNO₂ with MeONa give γ-nitro-β-phenyl-p-methoxybutyrophene, m. 92-3°; with I this yields the 2,2'-di-p-anisyl analog of II, dull blue needles or plates, m. 239-42°. 4-Methoxychalcone gives γ-nitro-β-p-anisylbutyrophene, m. 66° this yields the 4,4'-di-p-anisyl analog of II, needles with bright greenish metallic reflex, m. 288-90°. γ-Nitro-β-anisyl-p-methoxybutyrophene, an oil, yields 2,2',4,4'-tetra-p-anisylazadihydropyromethine, bluish coppery needles, m. 281-2°. p-Acetamidochalcone (34.4 g.) and 25.5 g. NaCN in 500 cc. MeOH, refluxed 20 min. with addition of 24 cc. AcOH in 65 cc. H₂O, give 12.2 g. of β-benzoyl-α-(p-acetamidophenyl)propionitrile, very pale yellow, m. 163-4.5°; heating with I for 10 min. at 180° gives the 4,4'-bis-(p-acetamidophenyl) analog of II, dull violet needles, m. about 370°. BzCH₂CH₂CHPhCN, on catalytic reduction

L20 ANSWER 236 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The effect of natural and artificial light on fibers and the humidity factor are discussed. Exposure of cotton to ultraviolet light alters the fluorescence, and increases the alkali-solubility and the absorption power for Fast Red Salt NB and Variamin Blue B. The formation of carboxyl groups and increase in reduction power are shown by the Cu number and other tests. The chemical changes are accompanied by a weakening of the fibers. The H₂O₂ formed by solar rays oxidizes alc. and aldehyde groups of cellulose to carboxyl groups and causes the formation of oxycellulose. Probably a splitting of the glucopyranose groups of the cellulose chain takes place. Wool is attacked at the S-S linkages of cystine and S is split off; a thio alc. is produced as well as the easily decomposed sulfenic acid, which is converted to several intermediate products and finally to H₂SO₄. The H₂SO₄ is best determined by Mease's method (C. A. 29, 932.8). Reactions of the basic groups and the increase of the ammonia N and the amine groups are discussed. The hydrolytic decomposition of wool is shown by its increased alkali and water solubility and lower strength and elongation. Even the wool of the living animal is damaged by the sun. Damaging effects are shown by certain unreduced vat dyes, such as Indanthrene Gold Orange 3G, and naphthol dyes, such as Naphthol AS-G. Complex oxidation reactions are responsible in which the dyes are reduced. Vat dyes that undergo this reaction are Cibacron Orange R, Indanthrene Brilliant Orange 3G and others. Oxides of Fe, Ti and Zn act as catalysts. Protective textile assistants Revatol S, Solidol N, Ludigol, Albatex BD, etc., fiber-weakening dyes should be avoided and finished fabrics should be protected with yellow or green light filters. Naphthols and amines protect by combining with O. Wool cat be protected by chroming. Fibers damaged by bleaching or alkali are damaged by sun more readily than those that have not been weakened chemically.

ACCESSION NUMBER: 1944:24015 CAPLUS
 DOCUMENT NUMBER: 38:24015
 ORIGINAL REFERENCE NO.: 38:3482b-g
 TITLE: The destruction of textile fibers by exposure to light
 AUTHOR(S): Salquain, J.
 SOURCE: Teintex (1942), 7, 275-81, 303-7
 From: Chem. Zentr. I, 1233(1943).
 CODEN: TEINAC; ISSN: 0040-2192
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 237 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 (Raney Ni) in MeOH or AcOEt, gives 95% of 2,4-diphenylpyrrole (V), m. 40°. Traces of III result on prolonged heating of V at 350°. With Se at 250° for 3 h., V gives 55% of III; on a large scale some MePhCHCH₂CH₂Ph was formed. Raney Ni at 350° (liq. phase) gives about 50% of III; in the vapor phase Ni-pumice catalyst gives 83% of III. The picrate and chloroplatinate dissociate on attempted crystn. The AcOH soln. of III becomes blue on heating. The concd. H₂SO₄ soln. is canary yellow and becomes violet on heating. III gives a blue color with the Ehrlich reagent. It couples with diazo compds. to give colored ppts. BzCH₂CH(C₆H₄OMe-p)CN with Raney Ni gives 2-phenyl-4-p-anisylpyrrole, b₇ 232-8°, m. 27° (picrate, pale yellow, m. 156-8°); Se at 250° gives 2-phenyl-4-p-anisylpyrrole (VI), m. 197-9°. p-MeOC₆H₄COCH₂CHPhCN gives 4-phenyl-2-p-anisylpyrrole, pale yellow, m. 74-5° (picrate, canary yellow, m. 180-1°); 4-phenyl-2-p-anisylpyrrole (VII), m. 205-7°. III in EtOH-HCl with NaNO₂ gives the 5-NO deriv. (VIII), green, m. 139-40°; HCl salt, orange-brown, m. 190° (decompn.); VI yields a 5-NO deriv. (IX), green, m. 176-7° (decompn.); HCl salt, red to transmitted light; VII yields a 5-NO deriv., the HCl salt of which (with 1 mol. MeOH), yellow, decompn. at 170°. VIII and III in AcOHAc₂O, heated on a steam bath for 0.5 h., give 95% of II. VI and the HCl salt of VIII in AcOH, refluxed 1 h., give 2,2',4-triphenyl-4'-p-anisylazadihydropyromethine, C₂₄H₁₈N₂, m. 256-7°; this also results from the HCl salt of IX and III. O₂NCH₂CHPhCH₂Ac and 3 parts HCONH₂, heated 0.5 h., give a brown product which does not resemble II. O₂NCH₂(CHPhCH₂Bz)₂ [m. 230° (decompn.)] and I or HCONH₂ gave no blue color. Chalcone and EtCH₂NO₂ give γ-nitro-β-phenylhexophenone in 2 forms, m. 156-8° and 88-90° (sepd. by the insoly. of the former in cold PhMe); neither form gave a colored product when heated with HCONH₂. γ-Nitro-β-phenylbutyrophene oxime, m. 108-10° (decompn. to a gum on standing); it readily gives the azamethine with I or HCONH₂. BzCH₂CHCH₂NO₂ with HCONH₂ gives a red-brown amorphous solid which is not related to the azamethines.

ACCESSION NUMBER: 1944:10121 CAPLUS
 DOCUMENT NUMBER: 38:10121
 ORIGINAL REFERENCE NO.: 38:1495d-1,1496a-h
 TITLE: 2,4-Diarylpyrroles. I. Synthesis of 2,4-diarylpyrroles and 2,2',4,4'-tetraarylazadihydropyromethines
 AUTHOR(S): Rogers, Maurice A. T.
 SOURCE: Journal of the Chemical Society, Abstracts (1943) 590-6
 CODEN: JCSAAZ; ISSN: 0590-9791
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 38:10121

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L20 ANSWER 239 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 ZKV → O.CH:CH.CH:CH2OCH2C:CH.CH:CH.O + H2O; (2) ZKV +
 O.CH:CH.CH:CH2C:CH.CH:CH.O + HCHO + H2O; (3) ZKV +
 O.CH:CH.CH:CH2C:CH.CH:CH.C(CH2-).O]n-2C:CH.CH:CH2C(CH2OH).O. Although these
 reactions explain the early steps in the resinification, they do not
 explain the complex mol. structure of the final resins. Against the idea
 that the resins are composed of a mass of mols. of the type in reaction
 (3) above, contg. small proportions of XIII and XIV are (1) the fact that
 the loss of wt. in the resinification of XV (taking into account the HCHO
 formed) is much less than that required by reaction (3) for large n
 values, and (2) the fact that the resins are not linear, sol. and fusible.
 but largely insol. and infusible. Probably in the resinification of XV,
 the initial reactions are analogous to those of II and HCHO, but the
 subsequent reactions are different in that the furan nucleus takes part in
 the reactions by reacting as a true unsatd. compd. through its double
 bonds, and thereby forming resins with tridimensional mols.

ACCESSION NUMBER: 1942:12306 CAPLUS
 DOCUMENT NUMBER: 36:12306
 ORIGINAL REFERENCE NO.: 36:1928e-1,1929a-1,1930a-1
 TITLE: The action of formaldehyde on ethyl pyromucate. II.
 Some resins of the furan series
 AUTHOR(S): Dinelli, Dino; Marini-Bettolo, G. B.
 SOURCE: Gazzetta Chimica Italiana (1941), 71, 117-28
 CODEN: GCITA9; ISSN: 0016-5603
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 240 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Very effective catalysts are produced by converting thio salts,
 such as the 5 compds. of Cu, Zn, Sb, As, Mo, W, Cr,
 Re, Ge, with heavy metal compds., such as halides, sulfates, nitrates,
 carbonates. The conversion can take place in aqueous or preferably organic
 solutions or suspensions. The two participating metals should preferably
 be different. Good solvents that can be used are acid amides, e. g.,
 formamide, acid chlorides, e. g., acetyl chloride, amino acids, ketonic
 acids, esters, nitriles, amines, aldehydes, ketones or nitro compds. In
 an example 0.5 mols. TiCl4, in C6H6 is slowly added to 1 mol. NH4
 thiocyanate in cyclohexylamine, while cooled by water. The
 brown precipitate is filtered and the solvents adhering to it are extracted
 with low-boiling solvents, e. g. acetone or alc. It is then
 treated with H2 with a slight admixt. of H2S at 300-400°. The
 resulting gray-black compound is pressed and used.

ACCESSION NUMBER: 1941:31431 CAPLUS
 DOCUMENT NUMBER: 35:31431
 ORIGINAL REFERENCE NO.: 35:4926h-1,4927a,4928a
 TITLE: Catalysts
 INVENTOR(S): Pier, Matthias; Simon, Walter; Jacob, Paul
 PATENT ASSIGNEE(S): I. G. Farbenindustrie AG
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 693985		19400627	DE	

L20 ANSWER 241 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB Lauronitrile treated with PhEtNLI produced 2-decyl-3-aminomyristonitrile,
 b3 230-5', which was hydrolyzed by concentrated H2SO4 to
 2-decyl-3-oxomyristamide, m. 114-15'. This amide with alc.
 KOH produced laurone. Hydrolysis of the β-imino nitrile with
 alc. HCl gave 2-decyl-3-oxomyristonitrile, m. 44-5'. With
 the same treatment stearonitrile produced 2-hexadecyl-3-
 iminoarachidonitrile, m. 54-5', and with H2SO4 2-hexadecyl-3-
 oxoarachidamide. Hydrolysis of the amide produced stearone. With
 alc. HCl 2-hexadecyl-3-oxoarachidonitrile, m. 68-9', was
 formed. Thiohydrolysis of appropriate nitriles gave: thiolaureamide, m.
 82-83'; thiomyrastamide, m. 87-8'; thiopalmitamide, m.
 93-4'; and thioleauramide, m. 96-7'. The Fries
 rearrangement of p-phenylphenyl stearate, m. 73-4', produced
 p-(p-hydroxyphenyl)stearophenone, m. 141-2', and
 2-hydroxy-5-phenylstearophenone, m. 63-4'. Methylation of these 2
 products with Me2SO4 gave p-(p-methoxyphenyl)stearophenone, m.
 116-17', and 2-methoxy-5-phenylstearophenone, m. 53-4'. On
 oxidation p-(p-methoxyphenyl)stearophenone gave terephthalic acid,
 identified by its di-Me ester. Stearonitrile and 2-ClOH7MgBr formed
 β-stearoylnaphthalene, m. 65-6'. Stearonitrile and p-PhC6H4Li
 gave p-phenylstearophenone, m. 108-9'. Stearonitrile and MeMgBr
 gave 2-nonacosanone, m. 55-6'. A Friedel-Crafts reaction between
 stearyl chloride and Ph2 or Ph2O gave p-phenylstearophenone or
 p-phenoxy-stearophenone, m. 62-3', resp. Sulfonation of
 p-phenylstearophenone formed 4-stearoyl-4-biphenylsulfonic acid, m.
 142-5', which on oxidation gave 4'-sulfo-4-biphenylcarboxylic acid
 (p-toluidine salt, m. 288-9'). Sulfonation of 4-PhC2H4CO2H also
 gave 4'-sulfo-4-biphenylcarboxylic acid which on fusion with KOH gave
 4'-hydroxy-4-biphenylcarboxylic acid. Decarboxylation yielded p-PhC6H4OH,
 identified by mixed m. p. With ClSO3H p-phenylstearophenone gave a
 trisulfonic acid. On oxidation 4'-sulfo-4-biphenylcarboxylic acid was
 obtained. Sulfonation of p-phenoxy-stearophenone gave 4-
 stearylphenylbenzenesulfonic acid, m. 95-8', which with dilute HNO3
 oxidized to 4-sulfophenoxybenzenesulfonic acid (p-toluidine salt, m.
 266-7'). Fusion with KOH gave p-HOC6H4CO2H. Hydrogenation of
 lauro- and stearonitriles with the Adkins Cu-Cr oxide
 catalyst gave didodecylamine, m. 52-3', and
 dioctadecylamine, m. 73-4', resp. Heating 1 mol of dodecyl
 chloride with 2 mol of didodecylamine produced
 tridodecylamine (HCl salt, m. 78-9'). Heating homologous
 compds. produced trioctadecylamine, m. 54-5' (HCl salt,
 m. 96-7'). 18-Iodopentatriacontane, m. 43.5-5', was obtained from
 18-pentatriacontanol. Laurone with dodecylmagnesium bromide gave
 12-dodecyl-12-tricosanol, b2 270-5', with octadecylmagnesium
 bromide, 18-octadecyl-18-pentatriacontanol, m. 58-9'. On treatment
 with P and I, 18-octadecyl-18-pentatriacontanol gave 18-iodo-18-
 octadecylpentatriacontane, m. 29-32', or by dehydration with
 p-MeC6H4SO3H gave 18-octadecylpentatriacontene, m. 42-4', which was
 possibly a mixture of olefins. Reduction of 18-iodo-18-
 octadecylpentatriacontane with Zn and HCl in AcOH produced
 18-octadecylpentatriacontane, m. 45-6'. No differences were observed
 between the preparation and reactions of these compds. and those of the
 other members of the same series having shorter chains.

ACCESSION NUMBER: 1940:41047 CAPLUS
 DOCUMENT NUMBER: 34:41047
 ORIGINAL REFERENCE NO.: 34:6220f-1,6221a-d
 TITLE: Reactions of some high-molecular-weight fat acid

L20 ANSWER 241 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
 derivatives
 AUTHOR(S): McCorkle, Miles R.
 SOURCE: Iowa State College Journal of Science (1939), 14, 64-6
 CODEN: ISCIJAF; ISSN: 0096-2783
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 242 OF 253 CAPLUS COPYRIGHT 2005 ACS ON STN
 AB 4,5,2-Me2(O2N)C6H2NH2 (I) condense with 1-arabinose and d-ribose in hot alc., with loss of 1 mol. H2O, to form orange-yellow crystalline products (C. A. 30, 464.4). Theoretically, these might be syn or anti forms of Schiff bases or α - or β -forms of furoid or pyranoid N-glucosides. On acetylation with Ac2O/C5H5N they smoothly yield tri-Ac derivs. which regenerate the original compds. with NH3-MeOH. Hence they are true N-glucosides. The condensation product of d-mannose and d-glucose with o-O2NC6H4NH2 and I likewise yield tetra-Ac derivs. almost quantitatively under mild conditions. The length of the O bridge was determined by the Helferich-Bredereck trityl method. It was first established that d-mannose 2-nitro-4,5-dimethylanilide (II), which undoubtedly has a free primary 6'-HO group, gives 90% of a trityl compound (III) with Ph3CCl in pyridine. The course of the tritylation can readily be followed quantitatively since, in the partition between water and benzene the o-nitroaniline glucoside remains entirely in the lower layer while the trityl compds. are readily soluble in benzene. d-Ribose 2-nitro-4,5-dimethylanilide (IV) and the 1-arabinose compound (V) yielded 88 and 82% trityl compds., resp., hence it is highly probable that they are furoid compds. The d-glucose compound (VI) and II are believed to be pyranosides, although without direct proof; d-glucose p-toluide, differing from VI only in having 1 NO2 and 1 Me group less, has been shown with certainty to be a pyranoid derivative (C. A. 30, 6334.5). The presence of an o-NO2 group decreases, to an extraordinary degree, the power of PhNH2 to condense. Whereas PhNH2, toluidine, phenetidine, etc., react smoothly with pentoses and hexoses on mere heating in alc., o-O2NC6H4NH2 under these conditions gives no trace of glucoside. In the earlier work on o-nitroaniline glucosides it was discovered by accident that a small amount of a catalyst (NH4Cl) is indispensable for their formation. As NH4Cl titrates in alc. with NaOH as free HCl, its action is evidently the same as that of free HCl in the Fischer synthesis of glucosides of alcs., but it has the remarkable property of catalyzing the condensation of the sugar with the solvent alc. much less than the reaction with the o-nitroanilines so that practically no O- and only N-glucosides are formed. With 1 mol. each of d-ribose and I and 0.05 mol. NH4Cl in alc., equilibrium is established at 0.60 mol. IV. With other pentoses and with o-O2NC6H4NH2 about the same equilibrium is reached. The yield of N-glucoside can be increased by (1) using an excess of I (the excess is recovered quantitatively on chromatographic separation of the products), or (2) using 1 mol. I and, after chromatographic separation of the glucoside, again boiling the residual product in absolute alc. with fresh NH4Cl. By either of these methods the yield can be increased to 80%. The water content of the alc. is very important. In 98% alc. only 30% IV was obtained after 4 hrs. heating of an equimol. mixture of the components and added NH4Cl but when, in a suitable apparatus, the alc. was continuously distilled into a 2nd flask with MeOH from which absolute alc. returned to the reaction flask, the yield arose to the usual 60%. On attempting to replace the NH4Cl by equivalent amts. of free HCl or PhNH2.HCl, decomposition (blackening) occurred. NaCl and (NH4)2SO4 proved ineffective. MeNH2.HCl had the same effect as NH4Cl (60% yield), while Me2NH.HCl and Me3N.HCl were less effective (25% reaction in the same length of time). The progress of the condensation can be followed quantitatively by the phase test (distribution of an aliquot between benzene and water). With hexoses, the yield obtainable seems to be less (40%). d-Mannose, because of its greater solubility in alc., is far better adapted than glucose to the

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 AB 163°. [α]_D20 160° ± 5° (MeOAc) d-Glucose
 2-nitroanilide, m. 70-5°, [α]_D20 26.1° ± 3°
 (pyridine); tetra-Ac deriv., m. 184°, [α]_D20 -75.2°
 ± 1° (MeOAc). 1-Arabinose 2-nitroanilide; tri-Ac deriv., m. 151°, [α]_D20 133.8° ± 1° (MeOAc).
 Triacetyl-d-xylose o-nitroanilide, m. 149°, [α]_D21 -109.5° ± 2° (MeOAc). VI, m. 214° (foaming), [α]_D21 11.7° (pyridine); tetra-Ac deriv., [α]_D22 -61.5° ± 0.5° (MeOAc). II, [α]_D20 -41.1° ± 1° (MeOAc); tetra-Ac deriv., m. 218°, [α]_D22 -93.8° ± 0.5° (MeOAc); trityl deriv., yellow prismatic rodlets with 1 H2O, m. 130° (decompn.). 6,7-Dimethyl-9-1-araboflavin, m. 310° (foaming), [α]_D25 -72.5° ± 2° (0.1 N NaOH), [α]_D22 442° ± 8° (0.4 M NaH2BO3); d-ribo compd. (lactoflavin), m. 290° (foaming), [α]_D22 -114° (0.05 N NaOH).
 ACCESSION NUMBER: 1937:130675 CAPLUS
 DOCUMENT NUMBER: 31:30675
 ORIGINAL REFERENCE NO.: 31:4324a-1, 4325a-1, 4326a
 TITLE: o-Nitroaniline glucosides
 AUTHOR(S): Kuhn, Richard; Strobele, Rudolf
 SOURCE: Ber. (1937), 70B, 773-87
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 31:30675

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 condensation. The o-nitroaniline glucosides sep. from abs. alc. in yellow prismatic needles; only VI and a 2nd modification (VII) of V formed orange-yellow rhombic leaflets. They all leave a strong bitter taste on the tongue. They are but little sol. in cold, easily sol. in hot water, but they soon begin to hydrolyze on heating. They are very sensitive to acids; 0.1 N HCl completely hydrolyzes them in 10 min. at 20°. The m. ps. are reproducible only when the conditions of crystn. and drying are controlled exactly, owing to the varying amts. of water of crystn. which can only with difficulty be completely removed without decompn. of the products; on heating, the glucosides are easily hydrolyzed by their own water of crystn. Moreover, rearrangements already seem to take during the m.-p. detns.; the m. ps. of the purest preps. often fell greatly after recrystn. (e. g., from 168° to 120°) without any hydrolysis (which can easily be detected by the phase test) occurring. The Ac derivs., readily obtained with Ac2O-C5H5N, are very well adapted to the characterization of the glucosides; they crystallize well, are sol. in CHCl3 and AcOEt, sep. without solvent of crystn., melt sharply, and have quite high rotations. Of the 2 compds. V and VII, V is obtained in far the larger amt. (95%); VII was isolated by virtue of its lesser soly. in alc. Both recrystallize unchanged from alc., give the same tri-Ac deriv., and are tritylated in pyridine, and therefore it is not impossible that they are the α - and β -forms. Mild hydrogenation of the o-nitroaniline glucosides to o-phenylenediamine glucosides is described elsewhere (following abstr.). On more vigorous hydrogenation 4 mols. H2 are taken up with simultaneous reduction of the nitro and the glucoside grouping to give the N-(polyhydroxyalkyl)-o-phenylenediamines (VIII) required for the synthesis of lactoflavins and similar flavins. The influence of H pressure, temp., solvent, concn., pH, etc., was detd. quantitatively in well over 100 expts. with different catalysts. The difficulty of the task was due less to the stability of the glucosidic O ring than to the facts that under the conditions of energetic reduction there occurs a hydrogenative cleavage to Me2C6H2(NH2)2 and that the benzene ring must not be hydrogenated. The desired purpose can be effected with Ni from Ni-Al alloys (Raney catalyst), with mixed Ni-Co-Cr catalysts, and with pure Ni preps. Addn. of Pt metals increases the activity in the desired direction. For the lab. prepn. of flavins are recommended Pt metals, especially Pd hydroxide pptd. on CaCO3 (Busch and Stove, C. A. 10, 2727) or on BaSO4 (prepn. described in detail). These are not the best catalysts but can always be easily prepd. of uniform quality. By means of them IV can be hydrogenated and then condensed with alloxan-H3BO3 to give 60% lactoflavin. Even more active is a catalyst prepd. by pptg. 1 atom Pd, 1/20 atom Zn and 1/80 atom Cu on CaCO3 (50 atoms Ca). The addn. of borate is also necessary to obtain a 60% yield of flavin. The o-nitroaniline glucosides form with H3BO3 complexes (as shown by the great increase in rotation) which are readily reduced to VIII. One mol. each of H3BO3 and NaOH are the optimal proportions; with only 0.5 mol. NaOH, the yield of flavin is decreased about 50%. The o-O2NC6H4NH2 and I were purified, after crystn. from benzene, by passing them in hot benzene through a column of Al2O3. d-V, lemon-yellow needles from alc., m. 111° (turning red) orange-yellow prisms with 1 H2O from water, m. 166°, [α]_D20 -20° ± 3° (pyridine); tri-Ac deriv., light yellow, m. 212°, [α]_D20 -137° ± 5° (MeOAc). 1-V, m. 111°, [α]_D18 26° ± 3° (pyridine); tri-Ac deriv., m. 212°, [α]_D25 139° ± 5° (MeOAc). VII, m. 166°, [α]_D20 76.0° ± 1° (pyridine). The tri-Ac deriv. of dl-V m. 213-14°, IV, m. 164°, [α]_D20 90° ± 3° (pyridine); tri-Ac deriv., m.

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 AB Acyl derivs. of dihydrofollicle hormone, described in 428,132 (below), are obtained by (1) treatment thereof with acylating agents, or (2) subjecting acyl derivs. of the follicle hormone to the action of reducing agents or to catalytic hydrogenation or (3) subjecting the follicle hormone to said reduction process in an acylating medium. The 1st process may be effected in a solvent in which the monoacyl derivative is insol. so as to yield the same, or the solvent may be so chosen that diacyl derivs. are obtained. The 2nd process yields monoacyl derivs. that may be further acylated. The hormones treated may be follicle hormone, C18H22O2, equilin, C18H22O2, or hippolin, C18H18O2, and the dihydrofollicle hormone may be such reduction products of any of these as contain a secondary OH group in the mol. but in which the C6H6 nucleus remains unchanged. Among examples, (1) the dihydrofollicle hormone in NaOH solution is treated with EtCl to yield a precipitate of the monobenzoyl compound, which is recrystd. from dilute alc., (2) the same starting material is dissolved in pyridine and treated with EtCl to yield the dibenzoyl derivative, which is precipitated on dilution with HCl, and (3) follicle hormone is dissolved in Ac2O together with AcONa and the reaction mixture, to which Zn dust has been added, is maintained at b. p. and then poured into H2O to yield a resinous precipitate of diacyldihydrofollicle hormone, which is recrystd. from dilute alc. In Brit. 427,561, Apr. 26, 1935, reduction products of the follicle hormone, having the formula C18H24O2 and which may be produced by the process of Brit. 428,132 (below), are further treated with catalytically activated H under such conditions that the C6H6 nucleus is hydrogenated. In an example, the initial reduction product is dissolved in alc. and treated with H at 160-170° in presence of a Ni-Cu catalyst. After the reaction is complete, H2O is added to precipitate the hydrogenated compound which is then separated by recrystn. from MeOH into crystalline and oily components, both of which are effective in the capon comb test. Cf. C. A. 28, 5182.7. In Brit. 427,588, Apr. 26, 1935, solns. of follicle hormone, C18H22O2, or of crude products containing, in addition, less saturated hormones, C18H20O2 and C18H18O2, are treated with H under pressure in presence of catalysts so as to saturate the C6H6 nucleus and to convert the keto group to the corresponding secondary alc. group. In examples, (1) a solution of the hormone in 10% KOH is hydrogenated at 100 atmospheric and 160° with use of a previously reduced Ni-Cu catalyst, the alkaline solution is extracted with Et2O and the extract is evaporated to dryness to yield a glass-like residue of formula C18H30O2, which may be distilled in a high vacuum to yield several crystalline fractions which are all active in the capon comb test. The alkaline mother liquors after the extraction with Et2O yield, on acidification, the dihydrofollicle hormone; (2) a solution of the hormone in cyclohexanol is hydrogenated at 60-70 atmospheric and 170° in presence of reduced Ni catalyst, the cyclohexanol is removed by vacuum distillation, the residue taken up in Et2O and the Et2O solution extracted with aqueous alkali solution to remove any unchanged starting material. Cf. C. A. 28, 5182.8. In Brit. 428,132, Apr. 26, 1935, reduction products of follicle hormones, where the keto group has been reduced to a secondary alc. group, are obtained by treatment of

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 the hormone with reducing agents under such conditions that the C6H6 ring is not hydrogenated. Starting materials may be the hormones of formulas C18H22O2, C18H20O2 or C18H18O2, the product always having the formula C18H24O2. Among examples, (1) hormone dissolved in alc. is treated with H at 20 atm. and 120° in presence of a Ni catalyst, isomeric products of m. ps. 168-170° and 198-202° being obtained, (2) hormone dissolved in wet AcOEt is treated with amalgamated Al foil, (3) crude hormone extd. from mares' urine is dissolved in AcOH, Na is added and the reaction stopped by passage of steam which drives off the AcOH; substances of hormone-like action obtained from vegetable materials, e. g., palm kernels, may be similarly treated. In Brit. 428,133, Apr. 26, 1935, reduction products of the follicle hormone, where the keto group has been converted to a secondary alc. group, are produced by reducing the oxime of the hormone to the corresponding amine and subjecting the latter to the action of a nitrite. In an example, a soln. of the oxime in alc. is treated with Na, dild. with H2O and evapd. to remove the alc., the mixt. is acidified with HCl, an aq. soln. of KNO2 is added, the mixt. is boiled and then extd. with Et2O, the ext. being evapd. to yield the dihydrofollicle hormone. In 428,215, Apr. 26, 1935, the mono- and di-acetates of dihydrofollicle hormone are prepd. by subjecting said hormone to the action of glacial AcOH in the presence of HBr, HI or toluenesulfonic acids as catalysts. In an example, the dihydrofollicle hormone, obtained as described in 428,132 (above) is dissolved in a glacial AcOH-HBr soln. (3:2), allowed to stand 2 days, poured into H2O, extd. with Et2O and purified by fractional crystn. from MeOH to yield the mono- and di-acetates.

ACCESSION NUMBER: 1935:51521 CAPLUS
 DOCUMENT NUMBER: 29:51521
 ORIGINAL REFERENCE NO.: 29:6707b-1,6708a-d
 TITLE: Sex hormones
 PATENT ASSIGNEE(S): Schering-Kahlbaum A.-G.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 427436		19350424	GB	

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 AB EtOH (I), iso-PrOH (II) and BuOH (III) have been mixed with air and passed over a number of catalysts with the production of aldehyde or ketone, acid, CO2, CO, saturated and unsatd. hydrocarbons and H. The oxidation of III resulted in the formation of a small quantity of ester with all catalysts and some HCHO with the mixed oxide catalysts. The following catalysts were used: Ag, Ni, Cu and Pt gauzes; Cu wire, Ag pellets, pellets of alloys of Cu and Zn, Ni, Bi, Pd, Pt, Au and Bi; oxide mixts. of U and Cu, V and Cu, Mo and Cu, V, Fe and Cu. Ag and Ag-Cu alloys produced better than 70% yields of aldehyde or ketone. I has been found to produce approx. 3 times as much CO2 as III and twice as much as II. The amount of H in the effluent gases was found to be small except when brasses were used as catalysts. Large quantities of Zn apparently promote dehydrogenation but not oxidation of the H. III was found to produce more unsatd. hydrocarbons than either I or II except with oxide catalysts, which caused the production of 10-15% of unsatd. compds. with iso-PrOH. This latter fact indicates that the oxide catalysts were not completely reduced to the metals. The production of saturated hydrocarbons is rather small in all other cases.

Acid production was found to be small and fairly constant for the same alcs. over different catalysts. The plated catalysts are unsatisfactory because the high temperature of reaction apparently caused solution of the plated metal into the supporting metal.

It also seems likely that the reaction takes place throughout the catalyst. Evidence for this view is found in the change in the mech. properties of the gauzes, wire and pellets and in the reduction of CuO to metallic Cu in the center of the pellets of a CuO-V2O3 mixture. The temperature of the reaction varied with the alc.-air ratios and with the catalysts employed. The Cu-V, Cu-Mo and Cu-V oxide mixts. were comparatively inactive and external heating was necessary. Heat was also applied to maintain a reaction at the surface of the 50% Zn-Cu alloy. The fact that small quantities of material incorporated into the metallic catalysts produce no marked effect indicates that a large portion of the surface of the catalyst is active in the catalytic oxidation of alcs.

ACCESSION NUMBER: 1928:18310 CAPLUS
 DOCUMENT NUMBER: 22:18310
 ORIGINAL REFERENCE NO.: 22:2140a-i
 TITLE: Catalytic oxidation with air of ethyl, isopropyl and butyl alcohols
 AUTHOR(S): Simington, R. M.; Adkins, Homer
 SOURCE: Journal of the American Chemical Society (1928), 50, 1449-56
 CODEN: JACSAT; ISSN: 0002-7863
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 AB Catalysts comprising base non-ferrous metals that form complex ions with NH3 are prepared by precipitating the oxides or hydroxides from solution in an excess of NH3 or an amine by removing the excess. The precipitate may subsequently be reduced. Suitable metals are Cu, Zn, Ag and Cd. Precipitation may be in the presence of suspended inert carriers, e. g., kieselguhr, or hydroxides that are not soluble in excess NH3, e. g., of Mn, Pb or Sn. The catalysts are particularly suitable for hydrogenation, e. g., of compds. containing a CO group, of nitro compds. and nitriles, the conversion of aldehydes and ketones to alcs., sugars to polyalcs. and acids and esters to the corresponding alcs. or hydrocarbons. They may also be used for dehydrogenation reactions.
 ACCESSION NUMBER: 1933:60817 CAPLUS
 DOCUMENT NUMBER: 27:60817
 ORIGINAL REFERENCE NO.: 27:5492e-g
 TITLE: Catalysts
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 392134		19330511	GB	

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 AB Vanillin benzyl ether (I) and MeNO2 gave 10% of a-3-methoxy-4-benzoyloxy-nitrostyrene (II) m. 122-3° with coucd. KOH as condensing agent but 89% using MeNH2. Reduction of II as usual with Zn and AcOH gave p-benzoyloxy-m-methoxy-phenylacetoxime (III), m. 118-9°. Reduce III in alc. and AcOH with Na-Hg at 30-50°, evaporate the alc., neutralize with NaOH and extract with Et2O. Addition of HCl to the dry Et2O solution gives p-benzoyloxy-m-methoxyphenylethylamine-HCl (IV), m. 173-5°. Free base b4 201-2°, m. 67-9°, picrate m. 171-2°, chloroplatinate m. 211° (decomposition); chloraurate m. 220-1° (decomposition). III must be purified before reduction to IV and the Na-Hg must be less than 3 times the theoretical. Addition of concentrated HCl to IV gives a smooth hydrolysis to β-vanillylethylamine-HCl (V), m. 210-1°; picrate m. 194-6°; chloroplatinate m. 211°; free base m. 156-7°. Acetylation of a-3-methoxy-4-hydroxynitrostyrene (VI) gives the 4-AcO derivative (VII). Reduction of VI

or VII did not give vanillylacetoxime but further treatment with Na-Hg gave 10% of V. Vanillin methoxymethyl ether, MeNO2, MeNH2-HCl and Na2CO3 gave a-3-methoxy-4-hydroxynitrostyrene-methoxymethyl ether, m. 102-3°. Reduction to the ethylamine was unsuccessful. I, CNCH2CO2Et, MeNH2.HCl and Na2CO3 in absolute alc. give Et α-cyano-β-p-benzoyloxy-m-methoxycinnamate (VIII), m. 127-9°. VIII in absolute alc. with KOH yields the K salt; Ag salt m. 185° (decomposition); free acid (IX) m. 202-3°. Reduction of IX with Na-Hg in H2O gives α-cyano-β-p-benzoyloxy-m-methoxyphenylpropionic acid (X), m. 125-6.5°. CSHN followed by NaOH converts X into p-benzoyloxy-m-methoxyphenylpropionitrile (XI) m. 78-9°. Reduction of XI in absolute alc. with Na gives γ-vanillylpropylamine (XII), and 3,4-MeO(Ph)CH2OC6H3CH2CH2CO2H (XIII). By addition of H2O evaporation of alc. and Et2O extraction unchanged XI is recovered. Acidify with HCl and extract with Et2O to isolate XIII. Evaporate the aqueous solution to dryness and extract with

absolute alc. for XII as the HCl salt, m. 154.6-5.6°. A further yield is obtained by addition of Et2O to the mother liquor. Chloroplatinate, m. 198-9° (decomposition); free base, m. 105-7°. Guaiacol condensed with CH2ClCOCl in the presence of AlCl3 in CS2 to a-chloroacetovanillone in poor yield. I refluxed with Ac2O and NaOAc gave p-benzoyloxy-m-methoxycinnamic acid (XIV) m. 188-90°. Na-Hg reduction of XIV yields XIII, m. 98.5-9.5°. Molten XIII and NH3 at 200-10°, gave the p-amide (XVI), m. 119-20°. Attempts to convert XVI to the thioamide and reduce it to the propylamine were unsuccessful. Reduction of vanillin oxime with Na-Hg and AcOH, gives vanillyl-methylamine (XVII); HCl salt m. 216-7°; picrate m. 198-200° (decomposition); with Zn dust and AcOH 16% of divanillyl-methylamine (HCl salt, m. 235-6°; free base, m. 134°), and XVII. p-Benzyl-m-methoxybenzaldoxime, m. 113-5°, by reduction in alc. AcOH by Na-Hg gives p-benzyl-m-methoxyphenylethylamine; HCl salt, m. 207-8°. 3,4-CH2OC6H3CH2Cl, KCN, Hg(CN)2 and H2O stirred at 76-8° for 4 hrs. gave an improved yield of piperonylacetoneitrile (XVIII), b. 160-6°. Hg(CN)2 and AgCN are positive catalysts; Cu(CN)2 is negative. In aqueous alc. 3,4-methylenedioxybenzyl Et ether, b8 118-20°, is formed in 25% yield with XVIII. Reduction of XVIII by Na in boiling absolute alc., gives β-piperonylethylamine (HCl salt, m. 208-9°, free base, b16 145°), and 3,4-CH2OC6H3Me, b. 193-5°, b14

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 83".
 ACCESSION NUMBER: 1928:11276 CAPLUS
 DOCUMENT NUMBER: 22:11276
 ORIGINAL REFERENCE NO.: 22:1345b-h
 TITLE: Synthesis of some fatty-aromatic amines containing phenolic hydroxy groups in the benzene nucleus
 AUTHOR(S): Kobayashi, Shozo
 SOURCE: Scientific Papers of the Institute of Physical and Chemical Research (Japan) (1927), 6, 149-65
 CODEN: SPIPAG; ISSN: 0020-3092
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 247 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB The following reactions have been found to be sensitized by ZnO in the presence of sunlight: (1) Decomposition of Fehling solution, (2) decomposition of cupriammonium oxalate, (3) formation of reducing sugars from CH₂O, (4) of reducing sugars from glycerol, (5) of Au from AuCl₃, (6) of Pt from PtCl₄, (7) oxidation of alcs. to the resp. aldehydes, (8) of quinine sulfate by H₂CrO₄, (9) of KI by K₂S₂O₈, (10) of Na citrate by I, (11) of H₂C₂O₄ by I, (12) of K₂C₄H₄O₆ by I, (13) of Na formate by I, (14) hydrolysis of maltose, (15) oxidation of iodoform, (16) of K₂C₄H₄O₆ by Br, (17) of NaNO₂ by I, (18) of HONH₂.HCl by I, (19) of N₂H₄.HCl by I, (20) of Na formate by HgCl₂, (21) of Na₂SO₃ by HgCl₂, (22) of N₂H₄.HCl by HgCl₂, (23) (NH₄)₂C₂O₄ and HgCl₂, (24) K₂C₄H₄O₆ and HgCl₂, (25) Na malate and HgCl₂, (26) K citrate and HgCl₂, (27) Na lactate and HgCl₂, (28) Cl₂CHCO₂Na and HgCl₂, (29) decomposition of H₂O, (30) of aqueous solns. of KMnO₄, (31) of K₂C₂O₄, (32) of aqueous solns. of K₂S₂O₈.
 ACCESSION NUMBER: 1928:7665 CAPLUS
 DOCUMENT NUMBER: 22:7665
 ORIGINAL REFERENCE NO.: 22:9151,916a-b
 TITLE: Zinc oxide as a general sensitizer for photochemical reactions
 AUTHOR(S): Bhattacharya, A. K.; Dhar, N. R.
 SOURCE: Quart. J. Indian Chem. Soc. (1927), 4, 299-306
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L20 ANSWER 248 OF 253 CAPLUS COPYRIGHT 2005 ACS on STN
 AB cf. S. C. A. 21, 3303. The dehydrogenating action of oxides which are practically unreducible by H was verified on compds, other than primary alcs. Passing piperidine vapors through an empty tube heated above 600° caused very little decomposition with very slight evolution of H; but when the vapors were passed under the same conditions through a tube containing MnO there was a steady evolution of H and production of a brown liquid consisting of a mixture of pyridine and dipyrindyl (due to dehydrogenation of the pyridine), boiling below 250°. The same dehydrogenation is readily obtained with reduced Ni even at 250°. As previously noted (loc. cit.), dehydrogenating oxides should also act as hydrogenation catalysts. PhNO₂ was entrained by an excess of H₂ through a pyrex tube containing MnO, which was heated progressively; at about 350° there was slight formation of H₂O vapor and NH₃; at 450°, and especially at 600°, the reaction was quite rapid, with evolution of considerable NH₃ and small quantities of CO₂, with condensation of a liquid containing NH₄OH, H₂O, PhNH₂, a little C₆H₆, and considerable proportions of Ph₂NH and Ph₃N. In the absence of catalyst, the decomposition is negligible. ZnO gives practically the same results as MnO. Al₂O₃ (prepared by precipitation and drying in the oven) gives but slightly more decomposition than when no catalyst is used. For direct hydrogenation of nitrous ethers ZnO is more active than MnO. With isoamyl nitrite the reaction proceeded regularly, giving mainly di- and tri-isoamylamine, and similarly with isobutyl nitrite. Direct hydrogenation of capronitrile (C₅H₁₁CN) proceeded similarly at 500° with either MnO or ZnO, with evolution of large quantities of NH₃ and formation of a mixture of the 3 corresponding hexylamines, the dihexylamine predominating. Hydrogenation over MnO gave unsatisfactory results with the ethylene hydrocarbons (with which reduced Cu frequently does not give good results) and with ketones (the rate of decomposition of the secondary alc. formed being much greater than the rate of formation at the active temperature of the catalyst).
 ACCESSION NUMBER: 1927:29377 CAPLUS
 DOCUMENT NUMBER: 21:29377
 ORIGINAL REFERENCE NO.: 21:35291,3530a-d
 TITLE: Catalytic hydrogenation and dehydrogenation by metal oxides
 AUTHOR(S): Sabatier, Paul; Fernandez, Antonio
 SOURCE: Compt. rend. (1927), 185, 241-4
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 AB cf. C. A. 17, 1479. Attempts under varied conditions and in different solvents to bring about a reaction between NH₃ and the vinyl side chain of quinine all failed and chlorohydroquinine likewise gave only quinine. Quinine chloride (I) reacted to only a minimal extent with aqueous or alc. NH₃, and the dibromide and tribromide proved equally resistant, no aminoquinine being obtained after heating many hrs. at 100° in sealed tubes with alc. NH₃ and Cu powder as catalyst. I heated with C₆H₄(CO)₂NH₂ and PhSO₂NH₂ (in vacuo to avoid the action of the air on the sensitive fused masses) at temps. above the m. p. of the higher melting component gives N-phthalyl-(II) and N-benzenesulfonylquinine-amine (III), resp., the reaction being complete in 16-20 hrs., as determined by measuring the amount of Cl split off. II is difficult to isolate owing to the ease with which it resinifies but forms a stable, easily purified picrate. Removal of the acyl group from II and III is very difficult because the resulting quinine-amine (IV) under the influence of the strong acids is apparently simultaneously demethylated and rearranged and a mixture of substances is obtained. Quinine bromide with C₆H₄(CO)₂NH₂ gives only an amorphous product. Neither the amorphous phenyl-(V) nor the crystalline p-bromophenylhydrazone of quinotoxine can be reduced with NaHg and AcOH, Na and EtOH or AmOH or Zn dust and AcOH to the quinotoxine-amine. II, prisms with 1H₂O, m. 140°, becomes yellow if dried by heating, is isolated through the picrate, yellow, m. 145°. Picrate of III, yellow, decomp. 137°. Sulfate of IV, sublimes about 180°, decomp. 227° in a sealed capillary, gives a positive thalleioquin reaction, dissolves in about 10 parts boiling H₂O. Picrate, canary-yellow precipitate, resinifies in all solvents on the gentlest warming, darkens and resinifies in the air, m. 120°. Di-Ac derivative, sirup, isolated as the dipicrate, which seps. in 2 forms, m. 122-5° (soluble in hot alc.) and 153° (insol. in alc., soluble in hot AcOH). Refluxed in excess of NaHCO₃ with Me₂SO₄ IV gives a H₂O-soluble quaternary NH₄ base isolated as the picrate, C₂₀H₂₂N₂O₂Me₂SO₄.H₂O, m. 115-20°, still contaminated by a green dye, which is formed during the methylation and cannot be completely removed with charcoal. Dipicrate of V, cinnabar-red crystalline precipitate, decomp. around 130-7°, gives V in brown amorphous form with dilute K₂CO₃.
 ACCESSION NUMBER: 1925:15770 CAPLUS
 DOCUMENT NUMBER: 19:15770
 ORIGINAL REFERENCE NO.: 19:2055c-h
 TITLE: Quinine-amine
 AUTHOR(S): Frankel, Sigmund; Tritt, Charlotte; Mehrer, Mathilde; Herschmann, Otto
 SOURCE: Ber. (1925), 58B, 544-54
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 GI For diagram(s), see printed CA issue.
 AB Much work on the hydrogenation of anthracene (I) is reported in the literature, but few of the compds. described have been obtained in pure form and the structure of no one has been proven beyond question. Reduction of I with Na or Na-Hg and alic. or liquid NH₃ or with HI and of anthraquinone with HI or with Zn and HCl gives the same dihydroanthracene (II), m. 108.5°, which has therefore been assumed to be the sym. meso- or 9,10-dihydro derivative β-Anthramine (III), which does not have the properties of a purely aromatic amine and is therefore to be represented by a formula containing a p-quinonoid grouping, rather than a true NH₂ group, yields on reduction a dihydroanthracene (IV) which is a true aromatic amine. This can be explained only by assuming that H is taken up at the meso-group and that IV has the structure C₆H₄(CH₂)₂C₆H₄NH₂. By diazotizing IV, reducing the product to the hydrazine and boiling this with CuSO₄, S. has obtained a product identical with II, thus definitely establishing its 9,10-dihydro structure. The structures of 2 of the tetrahydro and of the 2 decahydro derivs. of I described in the literature are unknown and those hitherto assigned to the 3rd tetrahydro and the hexahydro and octahydro derivs. are erroneous, while according to the Sachse-Mohr view (C. A. 13, 2661) a very large number of stereoisomeric tetradecahydro or perhydro derivs. (I of which is described in the preceding abstract) are capable of existence. The octahydro derivative octracene (VI) (or last 20 are possible) described in the preceding abstract probably has, from the way it is prepared, the structure C₆H₄(CH₂)₂C₆H₈ and it can also be obtained with extraordinary ease by reduction of I by the method described for C₁₀H₈ (Ger. pat. 325,721). Since, in the gentle reduction of I by different methods, II is always first formed, further catalytic reduction to V and also to tetracene (VI, below) must be accompanied by migration of H from the meso-group: C₆H₄(CH₂)₂C₆H₄ + C₆H₄(CH₂)₂C₆H₈ + C₆H₄(CH₂)₂C₆H₈. Proof of the structure of V was obtained by a progressive synthesis of V under conditions in which all rearranging influences were avoided: sym-octahydrophenanthrene (octanthrene) (VII) was similarly synthesized. Tetralin with ClCH₂COCl and a little P₂O₅ gives the isomeric 1- and 2-tetralyl chlorides (VIII and IX), which are easily separated IX condenses with CH₃Na(CO₂Et)₂ to di-Et-2-tetralylmalonate (X), converted by saponification and elimination of CO₂ into 2-tetralylpropionic acid (XI), which is reduced by Zn and HCl to 2-tetralylbutyric acid (XII), whose chloride on heating loses HCl and forms a mixture of 4-octanthrenone (XIII) and 1-octracenone (XIV), separated by means of their semicarbazones. XIV with Zn and HCl gives V and XIII yields VII. On the other hand, V on oxidation gives XIV and VII gives XIII. Although V differs slightly in its properties from the compound to which Godchot assigns the structure C₆H₄(CH₂)₂C₆H₁₀ (Contribution 'a l'acte. etude des hydrures d'anthracene, Paris, 1907), the identity of the 2 is established by their behavior on oxidation with CrO₃ and on bromination. G. bases the structure he gives his compound on the fact that with CrO₃ it gives a "hexahydroanthrone" (XV), whose benzylidene derivative on reduction gives a sec. alc., which by loss of H₂O yields a "benzylhexahydroanthracene" identical with that obtained by treating XV with PhCH₂MgCl and splitting off H₂O from the resulting tert. alc. As a matter of fact, however, the only property which the 2 hydrocarbons have in common is that both are oils at room temperature, and XV is really XIV. Indeed, almost all the compds. described by G. are erroneously formulated and his septl. results are also in part doubtful, to say the least, such as his statement that his octahydroanthracene gives with hot concentrated H₂SO₄ an unstable SO₃H acid

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 and amalgamated Zn in boiling HCl, b15 218-20°, m. 50-2°; with PCl₅ at 60-80° it yields a mixt. of the chloride and XIII + XIV (addn. of AlCl₃ to the chloride greatly diminishes the yield of ketones). The ketones cannot be isolated as the phenylhydrazones for these are converted by mineral acids into carbazoles: the mixt. of ketones boiled 5 min. in alc. with PhNHNH₂ in 3% AcOH gives the phenylhydrazone of XIV, yellowish, m. 115-8°, decomps. on standing and gives with boiling HCl 1,2-octracenophenocarbazole, m. 208°, which shows blue fluorescence in soln. and forms a black picrate, m. 144-5°; the mother liquors from the phenylhydrazones, boiled with HCl, give 4,3-octanthrenophenocarbazole, m. 142°; picrate, black-brown, m. 135-7°. The ketones can be sepd. as their semicarbazones, however, that of XIII, m. 229-31°, being sol. in 200, that of XIV, m. 250-1° (decompn.), in 500 parts boiling alc. XIV, from its semicarbazone with boiling 2 N HCl, b13 205-7°, m. 48°, gradually turns yellow in the air, is somewhat volatile with steam. The ketone mixt. refluxed with amalgamated Zn in HCl gives V, m. 73-4°, which with CrO₃-AcOH at room temp. yields unchanged V, XIV and a tarry mass from which can be isolated XVI; XIV is likewise obtained from V with Na₂Cr₂O₇, in dil. H₂SO₄ at 80°. The identity of XIV with the product obtained from XII and with Godchot's XV was established through the oxime, m. 147-8° (G., 143°), and the semicarbazone, m. 250-1°. 1-Benzylhexahydroanthracene (XXII), from PhCH₂MgI and subsequent distn. of the product (whereby H₂O is split off), viscous yellow fluid, b16 240-5°, forms no picrate, free picric acid, m. 120-2°, at once sepp. from the alc. or C₆H₆ soln. on concg.; in cold AcOH with Br it evolves HBr and H₂O ppts. a mixt. of a dibromide and a product poorer in Br (found, 30.4%), which on short warming in AcOH loses all its Br as HBr, giving 1-benzylanthracene, which no longer adds Br. XXII hydrogenated in the usual way begins to absorb H at 150° and the reaction is complete in 5 min., giving 1-benzyltetracene, viscous oil, b14 244-6°, which with 1 mol. Br₂ in AcOH gives a Br deriv., m. 106°. 2-Benzylidene-1-octracenone (XXIII), from V and BzH in alc., yellowish, m. 140°, reduced by Na in alc. to 2-benzyl-1-octracenol, m. 168-70°, which agrees in its properties with Godchot's "benzoyloctahydroanthranol" and with KHSO₄ at 170° yields 2-benzyl-Δ¹-hexahydroanthracene, b13 255-8°, i. e., 10-15° higher than the isomeric XXII; moreover, it shows a tendency to form a picrate (labile, to be sure), red leaflets from hot alc., m. 80-100°, soon dissociated in C₆H₆ with sepp. of free picric acid; with 1 mol. Br₂ in CS₂, also, it at once decolorizes the Br but evolves no HBr, forming a dibromide, m. 119-20° with evolution of HBr which, however, it loses only slowly in boiling AcOH. XXIII under 13 atm. takes up 3 mols. H₂ in a few min., giving 2-benzyltetracene, b12 248-51°, m. 65-6°. XVII, from XIV with Na and alc., m. 94-5° (Godchot, gives 81-2° for his "octahydroanthranol"); phenylurethan, m. 153° (G., 151-2°). XVIIII, m. 255°. Octracene-meso-sulfonic acid, from V and concd. H₂SO₄ at 70°, is isolated as the Na salt, leaflets with H₂O, losing H₂O in the air and sepp. with 1 AcOH when treated with Ac₂O; K, NH₄, Mg, Ca, Pb, Ba salts; chloride, from the Na salt and PCl₅ or directly from V with ClSO₃H, m. 87°; amide, m. 227-8°, forms a Na salt. The acid is quant. hydrolyzed by boiling concd. HCl; if its Na salt is used the free acid, which is difficultly sol. in concd. HCl, first seps. in leaflets with 3 H₂O and on further boiling hydrolyzes into V and H₂SO₄; fusion with KOH and treatment with Br also apparently split off the SO₃H group as H₂SO₄. The chloride with NaHSO₃ smoothly gives Na octracenesulfinate, which on further reduction yields the thiophenol. V

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 forming an easily sol. Ba salt and with KMnO₄ in Me₂CO or H₂O only phthalic acid. In the catalytic reduction of I it is difficult to det. when only 2 H atoms have been taken up but S. succeeded in obtaining at least a mixt. of di- and tetrahydro derivs., the 1st of which, identical with II, gives no picrate and is further reduced with astonishing rapidity to the 2nd (VI) and then to V. 1,2,3,4-Tetrahydroanthracene (VII), called tetracene in analogy with tetralin, forms a picrate and is oxidized by CrO₃ to tetracene quinone (1,2,3,4-tetrahydro-9,10-anthraquinone) (XVII), showing that it is a C₁₀H₈ deriv. VI is probably identical with Ipatiev's tetrahydroanthracene and Godchot's γ-compd., XVI probably being G.'s "dihydroanthranol." G.'s β-tetrahydroanthracene, on the other hand, was probably II contaminated with more highly reduced derivs. of I. Up to V, then, the hydrogenation of I proceeds without change in the common plane of the 14 C atoms of I; on further hydrogenation, however, which proceeds much less vigorously, the planes of the 3 rings must form an angle with each other, which will offer a certain resistance. The shifting of H atoms and bonds assumed above in the hydrogenation of I can probably also proceed in the reverse order, for 1-octracenol (XVIII), obtained by reduction of XIV, yields with dil. urea solns. 1-octracenylurea (XVIII), a replacement of a sec. alc. grouping characteristic of "enols" capable of desmotropism, so that XVII (as also 5-tetralol and 1-octanthrenol (following abstr.) very likely can assume the desmotropic form XIX. Again, XVI yields, as shown by Godchot, diacyl derivs. whose formation must be accompanied by a shifting of H atoms: C₆H₄(CO)₂C₆H₈ + C₆H₄(CO)₂CR + C₆H₈. When 3 parts pure I is reduced in a freshly prepd. mixt. of 2 parts tetralin and 0.07 part catalyst under 10-12 atm. of H hydrogenation begins at 120-50° and more than 2 atoms H are absorbed in a few sec. The product contains no unchanged I and with alc. picric acid gives the picrate of VI, the mother liquors of which yield II, m. 107-9°. IV on diazotization and treatment with SnCl₂-HCl gives β-hydrazinodihydroanthracene-HCl (the unstable free base decomps. on crystn. from alc.) which, immediately boiled with CuSO₄ and a little alkali, gives II. I absorbs more than 4 atoms H in 10 min. at 140-70°, giving 75% of the picrate, red-yellow, m. 116-7°, of VI, m. 103-5°, also obtained from II in the same way; di-Br deriv., faintly yellow, m. 166-8°. With KMnO₄ VI gives phthalic acid and with CrO₃-AcOH XVI, yellow, m. 155-5°, insol. in 2 N NaOH but dissolves with red-yellow color as the hydroquinol when gently warmed with Zn dust and is repptd. quant. by air. If the reduction of I is allowed to proceed until 8 atoms of H have been absorbed, it quant. gives V, b12 159-62°, m. 73-4°; if the reduction is allowed to proceed still longer there is also obtained a small amt. of a product b10 147-52°, having the compn. of a decahydroanthracene. Tetralin, ClCH₂COCl and P₂O₅, heated under a reflux, finally up to 190°, give IX, m. 63-4°, b17 202-3° (semicarbazone, m. 173°), and VIII, b0.2 (140-2°) (semicarbazone, m. 142-3°). IX boiled with alk. NaOCl gives 2-tetralic (tetrahydroanthracene-2-carboxylic acid, m. 151-2° X, from IX and CH₃Na(CO₂Et)₂ in boiling alc., gives on sapon. with alc. KOH, extn. with Et₂O and pptn. with HCl a tetracid K salt, C₁₀H₈O₄ (XX), crystg. unchanged from H₂O but sepp. from 85% alc. as the simple acid salt, C₁₀H₈O₅K, and then as the free acid (XXI), needles with 1 H₂O, which is obtained quant. by heating XX with HCl. XX splits off CO₂ quant. at 130-40°, yielding the acid K salt, C₂H₈O₃16K, m. 125°, of XI (obtained in the same way from XXI), m. 121-2°; Ag salt, γ-Tetralylcrotonolactone, ClOH11C₂CH₂CH₂CO₂O, from XI and boiling Ac₂O, m. 97-8°, smoothly adds 1 mol. Br in CS₂, is converted back into XI by hot NaOH, reduces NH₃-AgNO₃ to Ag in the cold. XII, from XI

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 in boiling H₂O with KMnO₄ gives pyromellitic acid, crystals with 2 H₂O, m. 264° (loss of H₂O); no phthalic acid could be detected. The pyromellitic anhydride, m. 286°, obtained by subliming the acid at 290° under 13 mm. gives with m-C₆H₄(OH)₂ and ZnCl₂ at 220° pyromellitein, red-yellow, very faintly fluorescent, converted by Br in C₆H₅N into pyromelliteosin, blue-red, dyeing silk in dil. AcOH contg. NaOC a deep blue-red. Pyromellitic diimide, from the NH₄ salt in NH₃ at 200°, dissolves in concd. H₂SO₄ without change and yields with hot NaOH Na pyromellitimide. The diimide with alk. NaOCl and subsequent pptn. with SO₂ yields p-aminoterephthalic (tereanilic) acid, light yellow, sol. in NaHCO₃ and in hot concd. HCl; di-HCl and H₂SO₄ salts, hydrolyzed by H₂O: Na salt, yellow powder; Cu salt, green-yellow ppt.; di-Et ester di-HCl salt, from the acid with alc. HCl, sol. in H₂O without hydrolysis, gives with NaHCO₃ the free ester, K₂Cr₂O₇-like crystals, m. 168°. ACCESSION NUMBER: 1925:9363 CAPLUS DOCUMENT NUMBER: 19:9363 ORIGINAL REFERENCE NO.: 19:1271f-i, 1272a-i, 1273a-i, 1274a TITLE: Hydrogenation of anthracene AUTHOR(S): Schroeter, G. SOURCE: Ber. (1924), 57b, 2003-24 DOCUMENT TYPE: Journal LANGUAGE: Unavailable

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AB 4-Nitro-2-naphthol is obtained by diazotizing 2,4-dinitro-1-naphthylamine with nitrosyl sulfate or NaNO₂ in concentrated H₂SO₄, pouring the diazo solution into H₂O to precipitate 4-nitronaphthalene-1-diazo-2-oxide, and heating this diazo-oxide with EtOH, alone or in the presence of a metallic catalyst such as Zn, Cu, Al or a Zn-Cu couple, or a reducing agent such as H₃P0₂. 2,4-Dinitro-1-naphthylamine is prepared either by heating 2,4-dinitro-1-naphthol with alc. NH₃ at 200°, or by dinitrating an acyl-α-naphthylamine or arylsulfonyl-α-naphthylamine, e. g., aceto-α-naphthalide and hydrolyzing the product, e. g., by heating with H₂SO₄. Azo dyes are obtained in substance or on the fiber by coupling diazo or tetrazo compds. with 4-nitro-2-naphthol; the following products are specified: a dark red wool dye from diazotized sulfanilic acid; and orange dye from diazotized p-nitraniline-o-sulfonic acid; a purple cotton dye from tetrazotized 4,4'-diaminostilbene-2,2'-disulfonic acid; a purple cotton dye from diazotized safranin; and azo dyes on the fiber from p-nitroaniline (bluish red), 2,4-dinitroaniline (red), dianisidine (dark blue), and primuline (reddish brown).

ACCESSION NUMBER: 1921:4306 CAPLUS
DOCUMENT NUMBER: 15:4306
ORIGINAL REFERENCE NO.: 15:7616-f
TITLE: Intermediate products; dyes
INVENTOR(S): Morgan, G. T.
PATENT ASSIGNEE(S): British Dyestuffs Corporation
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 152437		19190717	GB	

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property of coupling to form azo-compds. with the aforementioned polyhydric phenols in a mixt. of concd. HCl and AcOH. 4-Nitronaphthalene-1-diazo-2-oxide and resorcinol dissolved in glacial AcOH containing HCl while standing 2 days, interacted, giving B reddish black nodules, M. 254-6°. It gave an intense violet color with concd. H₂SO₄ and on unordanted wool it furnished red-brown shades, becoming violet on treatment with CuSO₄ and AcOH. On Cr-mordanted wool a blue-black lake was produced. When A was coupled with phloroglucinol or 1,3C10H₆(OH)₂ in HCl or AcOH similar hydroxyazo compds. were formed which dyed Cr-mordanted wool violet-blue shades. They showed little tendency to crystallize. A, when heated in alc. with Cu, gave off AcH and tests showed the presence of a phenolic compd. After removing the alc. and extg. with water, the cold water soln. gave yellow needles, C (38% yield). Zn dust was more effective than Cu, giving a 45% yield and Al gave 58-64%. Using 30% H₃P0₂ the yield of C was 53%. Without any metallic catalyst a yield of 45% was obtained. C crystd. from water, CCl₄, light petroleum in yellow needles, and as red prisms from C₆H₆. It gave a red soln. with aq. alkali hydroxides and its K salt formed red glistening needles. With Sn and HCl, C was reduced to 2,4-C10H₆(OH)₂ and finally to 1,3-C10H₆(OH)₂ (Ber. 28, 1952(1895)). C was not converted into 2,4-C10H₆(NH₂)NO₂ with aq. or alc. NH₃. The Na salt of C in dry xylene with Me₂SO₄ gave 4-nitro-o-naphthyl methyl ether, brown needles, m. 100-3°, from C₆H₅SO₂H. With PhN₂Cl C in NaOH gave the insol. dark red benzene-2-azo-4-nitro-β-naphthol, which from C₆H₆ came down in needles, M. 206-7°. This was only sparingly sol. in alc. and in aq. NaOH it was insol., showing that the NO₂ group had not rendered the compd. acidic. It gave a magenta color with concd. H₂SO₄ and alc. NaOH gave a deep red soln. p-O₂NC₆H₄N₂Cl and C in NaOH gave a purplish ppt. of p-nitrobenzene-1-azo-4-nitro-β-naphthol. From glacial AcOH it sepd. in nodules, m. 231°, insol. in aq. alkali hydroxides but in alc. NaOH it gave an intense purple-blue color. Concd. H₂SO₄ and the azo compd. gave a purple color. When developed on calico padded with alk. C and Turkey red oil, this azo compd. gave a crimson-red quite distinct from p-nitroaniline red. When C in NaOH was treated with dil. 2,4-dinitrobenzenediazonium sulfate, a dark reddish brown ppt. appeared, azo-4-nitro-β-naphthol, nodular needles from anisole, m. 132-166°. A second product was also prepd., 1-[2,4-dinitrophenylazo]-4-nitro-β-naphthol, dark red tabular crystals from anisole, and this compd. with its 3 NO₂ groups was apparently acidic. The 4-NO₂ deriv. of orange II was prepd. by adding diazotized sulfanilic acid to C in NaOH. The dye was salted out as a red powder which dyed wool deep red shades. C with 4,2-O₂N(HO₃)C₆H₃NH₂ gave an acid azo dye which gave orange shades on wool. Diazotized picramic acid with C gave a dye, m. 250°, which gave red-brown shades on unordanted wool, the Cr lake being brownish red. Calico padded with C with aq. Na₂CO₃ and Turkey red oil and passed through NaOAc soln. and the bisdiazonium chloride from dianisidine was dyed dark blue. Primuline dyed and diazotized on cotton with C gave a purple-maroon color and direct cotton colors dyeing in purple were obtained by coupling C with the diazo deriv. of safranin and [2,4-HO₃(H₂N)C₆H₃CH₂]₂. The diazotization of 2,4-dinitroaniline sulfate. The 2,4-(O₂N)C₆H₃NH₂ was obtained in quant. yield by heating 2,4-(O₂N)C₆H₃Cl in an autoclave with aq. NH₄OH. It was diazotized in concd. H₂SO₄ by adding dry NaNO₂ and after 2 hrs. pouring the mass into ice water. 2,4-Dinitrobenzenediazonium sulfate was present since, when β-naphthol was added, 2,4-dinitrobenzenediazo-β-naphthol was deposited as orange-red needles. The product crystd. from glacial AcOH did not m. 270°. The elimination of the o-NO₂ group in 2,4-dinitrobenzenediazonium sulfate was effected by pouring the diazo

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GI For diagram(s), see printed CA Issue.
AB Earlier work by Morgan and Porter (C. A. 9, 2061) on the nitrated o- and p-diazo oxides of the C₆H₆ series showed that 4-nitrobenzene-1-diazo-2-oxide obtained by diazotizing 5,2-O₂N(H₂N)C₆H₃OH has the property of forming dihydroxyazo deriva. which yield metallic lakes having distinctive colors, and the corresponding diazo-oxides of C10H₈ are now reported and comparison is made of the differences manifested between the benzenoid diazo-oxides and their analogs in the C10H₈ series. 4-Nitronaphthalene-1-diazo-2-oxide (A) (Friedl-ander, Ber. 28,1951(1895)) has not been examined hitherto in detail. It couples with the more reactive phenols, resorcinol, phloroglucinol, and 1,3C10H₆(OH)₂ and the formation of azo compds. takes place in alkaline solution and also with greater facility in concentrated mineral acid solution. 4-Nitro-o-naphthol-1-azoresorcinol (B) is an acid mordant dye giving reddish brown shades on wool which are changed into lakes of characteristic color by the action of metallic mordants (Cr, Cu, etc.). It loses its diazo group and yields 4-nitro-β-naphthol (C), the fourth compound of 7 possible nitro-naphthols. This is less reactive than β-naphthol, less easily alkylated and couples more slowly with azo compds. The 4-nitro-1-azo-β-naphthols from PhN₂Cl and p-O₂NC₆H₄N₂Cl are insol. in aqueous alkaline hydroxides, resembling azo-β-naphthols, the insol. of which in aqueous alkalis is a matter of great industrial and theoretical importance. 1,2,4C10H₅(NH₂)(NO₂)₂ (D), the starting point of the compds. of the investigation, was obtained in 3 ways. By the 1st method α-naphthol was added to concentrated H₂SO₄ and the mixture was stirred till a clear solution was obtained. On heating and then cooling, α-naphtholdisulfonic acid separated. A water solution of this solid was added to HNO₂ kept below 10°. The reaction mixture was then warmed to 100° and after cooling 1,2,4-C10H₅(OH)(NO₂)₂ separated. The latter and alc. NH₃ were heated in an autoclave and on cooling D separated. By the 2nd method α-C10H₇NHAc (Meldola, Ber. 19; 2683(1886)) in glacial AcOH was nitrated below 70°, then at 96°, when 2,4-(O₂N)C10H₅NHAc separated. This was added to water and H₂SO₄ and after warming the mixture was added to ice water, when D precipitated. By the 3rd method, starting with α-C10H₇NH₂ mixed with p-MeC₆H₄SO₂Cl and anhydrous NaOAc, MeC₆H₄SO₂NH₂OH was obtained. This was dissolved in glacial AcOH and nitrated. On cooling, toluene-p-sulfonyl-2,4-dinitro-β-naphthylamide separated in radiating pale yellow needles, m. 165°. When this was hydrolyzed by H₂SO₄ D was obtained. A mixture of D, concentrated H₂SO₄ and nitrosyl sulfate was treated with ice and then poured into water. The precipitated diazo-oxide, crystallized from petr. ether in yellow needles, m. 131-2°. The diazotization was also effected with dry NaNO₂ in concentrated H₂SO₄. The diazo-oxide was prepared from the acyl derivative of D without isolating the base itself. (O₂N)C10H₆NH₂SO₂C₆H₄Me in H₂SO₄ was hydrolyzed by warming to 70° and after cooling to 0° NaNO₂ in H₂SO₄ was added. From the dilute mixture the diazo-oxide separated. This is 4-nitronaphthylene-1-diazo-2-oxide. It is sparingly soluble in water but readily soluble in C₆H₆, Me₂CO, AcOH, and moderately soluble in alc. On adding NaOH to the alc. solution H₂ was evolved. AcH was produced also. With resorcinol, phloroglucinol, or 1,3-C10H₆(OH)₂ the diazo-oxide in alc. on addition of one drop of NaOH gave a blue color which may be used as a test, and the diazo-oxide has the remarkable

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soln. into ice water containing Na₂CO₃. A yellow ppt. sepd., m. 64-80°. When coupled with alk. β-naphthol it gave a purple-brown azo compd. The yellow alk. soln. was added to an excess of alk. β-naphthol and a deep blue sparingly sol. Na salt sepd. which with alc. HCl gave a purple azo-β-naphthol, m. 228°, identical with the 4-nitro-2-hydroxybenzenediazo-β-naphthol obtained from the internal diazo-oxide of 5,2-O₂N(H₂N)C₆H₃OH (C. A. 9, 2061). The production of a di-HO compd. from β-naphthol and the alk. soln. of the diazo deriv. of 2,4-(O₂N)C₆H₃NH₂ showed replacement of the o-NO₂ group by OH as is confirmed by the following expt. Diazotized 2,4-(O₂N)C₆H₃NH₂ was treated with resorcinol in aq. NaOH and the intensely purple-red soln. on heating yielded after salting a Na salt which with H₂SO₄ gave the orange-red 5-nitrophenolazo-2-resorcinol, small red needles from glacial AcOH, decomp. 225°. On unchromed wool, it gave orange-red shades, changing to deep claret on subsequent chroming. The marked alteration of shade produced by mordanting with Cr suggested the rearrangement of two HO groups contiguous to the azo group. Further evidence was obtained by a comparative expt. made on 2,6-C10H₆(OH)₂SO₃H. Consequently, an acid soln. of 2,4-dinitrobenzenediazonium sulfate was added to Shaffer's salt in NaOH and after salting, 2,4-dinitrobenzenediazo-β-naphthol-6-sulfonic acid sepd., which gave orange shades on unordanted wool not changed by chroming. 2,4-(O₂N)C₆H₃NH₂ diazotized as before and rendered alk. with aq. Na₂CO₃ was added to an alk. soln. of Shaffer's salt. On salting out, 4-nitro-2-hydroxybenzenediazo-β-naphthol-6-sulfonic acid sepd. This gave reddish brown on wool, changing to violet on chroming. (O₂N)C₆H₃NH₂, diazotized as before in acid soln., was treated with CuSO₄ and the liquid was rendered alk. with Na₂CO₃ and added to an alk. soln. of Schaffer's salt. The dye which was salted out gave purple on wool, not changed by after-chroming. These expts. point conclusively to the formation of a sol. form of 5-nitro-2-diazophenol when an acid soln. of 2,4-dinitrobenzenediazonium sulfate is made alk. with Na₂CO₃. The azo dyes formed with the various phenol deriva. and reactive amines all form distinctively colored metallic lakes. A table of azo dyes from 5-nitro-2-diazophenol is given. The azo coloring matters when dyed on unordanted wool are extremely sensitive to metals, a property due doubtless to the ease with which the metallic lakes are formed.

ACCESSION NUMBER: 1920:3899 CAPLUS
DOCUMENT NUMBER: 14:3899
ORIGINAL REFERENCE NO.: 14:733e-1,734a-1,735a-1
TITLE: Constitution of internal diazo oxides (diazophenols). II.
AUTHOR(S): Morgan, Gilbert T.; Evans, Eric D.
SOURCE: Journal of the Chemical Society, Abstracts (1919), 115, 1126-40
CODEN: JCSAAZ; ISSN: 0590-9791
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB The formation of arylanthranilic acids from o-chlorbenzoic acid and aromatic amines was shown to be due to the presence of minute amounts of inorganic catalysts, whose efficiency decreased in the order of the salts of copper, iron, nickel, zinc, lead, and platinum chloride, the latter having no influence. Increasing the amount of the catalyst also increased the velocity of the reaction. o-Brom. and o-iodbenzoic acid apparently condensed just as well in the absence of a catalyst. In all the cases, the yields were increased if amyl alcohol or nitrobenzene was added. The arylanthranilic acids were used for the preparation of acridone derivatives (by heating with concentrate H2SO4 or with PCl5 and then AlCl3)

and of unsym. diphenylamine derivatives (by heating alone). The fluorescence of the acridone derivatives was also studied. Experimental.

ACCESSION NUMBER: 1908:293 CAPLUS
DOCUMENT NUMBER: 2:293
ORIGINAL REFERENCE NO.: 2:87h-1,88a-g
TITLE: On Arylanthranilic Acids
AUTHOR(S): Ullmann, Fritz
CORPORATE SOURCE: Techn. Hs., Berlin
SOURCE: Ann. (1908), 355, 312-58
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 2:293

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COST IN U.S. DOLLARS

SINCE FILE

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SESSION

FULL ESTIMATED COST

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SINCE FILE

TOTAL

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SESSION

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